

Off-target Movement Assessment of a Spray Solution Containing  
MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

<b>TEST SUBSTANCE:</b>	MON 76980
<b>DATA REQUIREMENT(S):</b>	US EPA OPPTS 835.8100: Field Volatility US EPA OPPTS 840.1200: Spray Drift Field Deposition
<b>AUTHOR(S):</b>	Will Griesse
<b>STUDY COMPLETION DATE:</b>	April 3, 2020
<b>SPONSOR:</b>	Monsanto Company <sup>1</sup> 700 Chesterfield Parkway West Chesterfield, MO 63017
<b>PRIMARY TEST FACILITY MANAGEMENT:</b>	Rodrigo Sala, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO
<b>LABORATORIES:</b>	Lange Research and Consulting, Inc 4746 W. Jennifer Ave. Suite 105 Fresno, CA 93722
Exponent, Inc. 1800 Diagonal Road, Suite 500 Alexandria, VA 22314	Eurofins EAG Agrosience, LLC 7200 East ABC Lane Columbia, MO 65202
Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017	AGVISE Laboratories 604 Highway 15 West P.O. Box 510 Northwood, ND 58267
<b>MONSANTO STUDY ID:</b>	REG-2019-0035
<b>REFERENCE NUMBER:</b>	TRR0000087
<b>LANGE STUDY ID:</b>	LR19397
<b>EUROFINS STUDY ID:</b>	89311
<b>AGVISE STUDY ID:</b>	19-1371, 19-115


**Total pages: 633**

---

<sup>1</sup>Monsanto is an indirect, wholly-owned subsidiary of Bayer.

## NO CLAIM OF CONFIDENTIALITY

No claim of confidentiality, on any basis whatsoever, is made for any information contained in this document. I acknowledge that information not designated as within the scope of FIFRA sec. 10(d)(1)(A), (B), or (C) and which pertains to a registered or previously registered pesticide is not entitled to confidential treatment and may be released to the public, subject to the provisions regarding disclosure to multinational entities under FIFRA 10(g).

Company:	<u>Monsanto Company</u>
Company Agent:	<u>Thomas B. Orr</u>
Title:	<u>Regulatory Affairs Manager</u>
Signature:	<u></u>
Date:	<u>03 Apr 2020</u>

## SUBMISSION AND USE OF MATERIALS UNDER FIFRA

The inclusion of this page is for quality assurance purposes and does not necessarily indicate that this study or document has been submitted to the United States Environmental Protection Agency (US EPA). The text above applies only to use of the data or document by the US EPA in accordance with the provisions of the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA), and not to any other use or use by any other agency or government.

We submit this material to the US EPA specifically under the requirements set forth in FIFRA as amended, and consent to the use and disclosure of this material by US EPA strictly in accordance with FIFRA. By submitting this material to US EPA in accordance with the method and format requirements contained in PR Notice 2011-3, we reserve and do not waive any rights involving this material, including but not limited to copyright and data compensation, that are or can be claimed by the Company notwithstanding this submission to the US EPA.

## GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This study was conducted in accordance with the United States EPA Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) Good Laboratory Practice (GLP) Standards (40 CFR 160), with the following exceptions:

1. Test site observations such as slope estimates
2. Pesticide and crop history for the test plot
3. Soil taxonomy data provided by the USDA Natural Resources Conservation Service (NRCS)
4. Test plot preparation prior to application
5. SpotOn nozzle calibration devices used to calibrate the sprayer not maintained under GLP and no SOP in place SpotOn calibrators were verified prior to use Verification was documented in the raw data
6. GPS Coordinates.
7. Study weather data from external sources
8. Indirect flux and off-target air concentration and vapor deposition modeling was conducted by Exponent Inc , which is a non-GLP facility Exponent Quality Policy can be found in the Exponent sub-report
9. Light Detection and Ranging (LIDAR) elevation map displayed in the Plant Effects Sub-Report



Will Griese  
Study Director  
Monsanto Company

4/3/2020

Date



Thomas B. Orr  
Sponsor Representative  
Monsanto Company

03 Apr 2020

Date



Submitter  
Monsanto Company

03 Apr 2020

Date

## QUALITY ASSURANCE STATEMENT

**Study Title:** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

**Study No.:** REG-2019-0035

Monsanto Company Quality Assurance Unit (Monsanto QAU) was Lead QA oversight for this study. Reviews conducted by Monsanto Quality Assurance confirm that the final report accurately describes the methods and standard operating procedures followed and accurately reflects the raw data for the portion of the study conducted by Monsanto Company. This confirmation excludes the following data:

- \* Indirect flux and Off-target Modeling from Exponent. Exponent is not a GLP facility.

Reviews conducted by Eurofins EAG Agrosience, LLC are enclosed within their analytical sub-report and are specified on their individual QA Statement.

Reviews conducted by Lange Research and Consulting, Inc and those outsourced by Lange to Knipp Consulting LLC and ASK GLP Consulting are enclosed within their field sub-report and are specified on their individual QA Statement. Included in the field sub-report QA Statement are reviews conducted by AGVISE Laboratories. Below is a corrected list of inspections and dates reported to study director and management for AGVISE inspections based on documentation provided to the Lead QA.

Dates of Inspection/Audit	Phase	Date Reported to Study Director/Management
7/19/2019	AGVISE Procedure Audit - pH of Water	8/7/2019
7/30/2019	AGVISE Procedure Audit – pH analytical Procedure	8/7/2019
8/6/2019	AGVISE – Raw Data Audit (19-1371)	8/7/2019
8/7/2019	AGVISE – Raw Data Audit (19-115)	8/7/2019

Following is a list of reviews conducted by Monsanto Regulatory Quality Assurance on the study reported herein. An additional Monsanto Regulatory Quality Assurance audit of Flux data and Modeling Sub-report is specified within the AD and IHF modeling sub-report on the QA Statement.



Dates of Inspection/Audit	Phase	Date Reported to Study Director/Management
6/30/2019	0 DAT plant effects measurements	7/3/2019
7/30/2019	28 DAT plant effects measurements	7/30/2019
7/29/2019	Sample Preparation and Mass Spec Analysis – filter paper	8/13/2019
8/23/2019	Data Audit – filter paper analysis	9/6/2019
1/22/2020	Statistics Data and Report Audit – Spray Drift	2/5/2020
2/7/2020	Analytical Report Audit	2/21/2020
2/20/2020	Statistical Data and Report Audit – Plant Effects	2/20/2020
3/23/2020	Report and Data Audit – Plant Effects Sub-report	3/26/2020
3/25/2020	Final Report and Data Audit	3/29/2020



Carrie L. Logan, M.S., M.B.A  
Quality Assurance Team Lead, RQAP-GLP  
Monsanto Regulatory, Monsanto Company

4/3/2020

Date

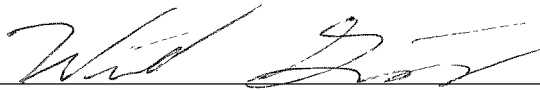
## REPORT APPROVAL

**Report Title:**

Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

This report is an accurate and complete representation of study activities.

**Study Director:**



---

Will Griese  
Regulatory Field Studies Monitor  
Bayer CropScience

4/3/2020

---

Date

## STUDY INFORMATION

### Contributors

The following personnel contributed to the conduct of this study and the final report in the capacities indicated:

#### Name:

#### Role:

#### **Monsanto Company**

Will Griese

Study Director, Plant Effects Data Collection

Thomas B. Orr, M.S.

Sponsor Representative

Rodrigo Sala, Ph.D.

Testing Facility Management

Michael Shepard, Ph.D.

Analytical Chemistry Principal Investigator, Author

Naresh Pai, Ph.D.

Modelling Principal Investigator-Volatility

Tim Fredericks, Ph.D.

Field Research Principal Investigator – Plant Effects

Adam Schapaugh, Ph.D.

Statistical Analyses Principal Investigator – Plant Effects and Deposition

Amanda Chen, Ph. D

Analytical Phase, Author

Brian Schaefer

Analytical Phase, Study Personnel

Erik Sall, Ph.D.

Modelling Phase, Author

Keguo Huang, Ph.D.

Plant Effects and Deposition Modelling Statistician

Steven Voss

Plant Effects Data Collection

Lance Schuler, Ph.D.

Plant Effects Data Collection

Mark Thomas, Ph.D.

Plant Effects Data Collection

Brian Krebel, M.S.

Plant Effects Data Collection

#### **Lange Research and Consulting, Inc.**

Alex Gibbs

Field Research Principal Investigator – Volatility and Deposition

Jay Hoy

Plant Effects Data Collection

#### **Exponent, Inc.**

Richard Reiss, Sc.D.

Modelling Principal Investigator-Volatility, Author

Jelena Popovic

Modelling Phase, Author

#### **Test Site**

Chris Schaubert

Field Cooperator

#### **AGVISE Laboratories**

Larry Wikoff

Testing Facility – Soil and Water Characterization  
Principal Investigator

**Eurofins EAG Agrosience, LLC**

Matthew Rebstock

Testing Facility – Analytical Chemistry Principal  
Investigator

Bryanne Cornine

Analytical Phase, Study Personnel

Wesley Fain

Analytical Phase, Study Personnel

Courtney Travis

Analytical Phase, Study Personnel

**Study Dates**

Study Initiation Date: May 31, 2019

Experimental Start Date: July 2, 2019

Experimental Termination Date: August 6, 2019

Study Completion Date: April 3, 2020

**© 2020 Bayer Group. All Rights Reserved.**

*This document is protected under national and international copyright law and treaties. This document and any accompanying material are for use only by the regulatory authority to which it has been submitted by Monsanto Company and its affiliates, collectively "Bayer Group", and only in support of actions requested by Bayer Group. Any other use, copying, or transmission, including internet posting, of this document and the materials described in or accompanying this document, without prior consent of Monsanto Company, is strictly prohibited; except that Monsanto Company hereby grants such consent to the regulatory authority where required under applicable law or regulation. The intellectual property, information and materials described in or accompanying this document are owned by Bayer Group, which has filed for or been granted patents on those materials. By submitting this document and any accompanying materials, Monsanto Company and the Bayer Group do not grant any party or entity any right or license to the information, material or intellectual property described or contained in this submission.*

**Retention of Raw Data**

The protocol, raw data, documentation, records, data supporting the analytical and statistical analyses conducted by Monsanto and the final report for this study are archived at Monsanto Company. Additional data, or copies will be archived or retained as follows:

- Eurofins EAG Agrosience, LLC: Analytical report and raw data were transferred to Monsanto Company archives prior to completion of the study.
- Exponent, Inc.: Modelling input and supporting data were transferred to Monsanto Company and are archived at Monsanto.
- Lange Research and Consulting, Inc.: Field report, field notebook and raw data have been archived and the Lange facility in Fresno, CA, pending archive transfer to Monsanto.
- AGVISE Laboratories: Soil and Water characterization reports and supporting data are archived with AGVISE.

## TABLE OF CONTENTS

NO CLAIM OF CONFIDENTIALITY .....	2
SUBMISSION AND USE OF MATERIALS UNDER FIFRA .....	2
GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT .....	3
QUALITY ASSURANCE STATEMENT .....	4
REPORT APPROVAL .....	6
STUDY INFORMATION .....	7
TABLE OF CONTENTS .....	10
ABBREVIATIONS AND SYMBOLS .....	12
<b>1.0 INTRODUCTION .....</b>	<b>13</b>
<b>2.0 STUDY OBJECTIVE .....</b>	<b>14</b>
<b>3.0 STUDY SUB-REPORTS .....</b>	<b>15</b>
<b>3.1 Field Sub-Report .....</b>	<b>15</b>
<b>3.2 Analytical Sub-Reports .....</b>	<b>15</b>
<b>3.3 Volatile Flux Estimation Sub-Report .....</b>	<b>16</b>
<b>3.4 Indirect Flux and Off-Target Modeling Sub-Report .....</b>	<b>16</b>
<b>3.5 Spray Drift Deposition Modeling Sub-Report .....</b>	<b>16</b>
<b>3.6 Plant Effects (Drift and Volatilization) Sub-Report .....</b>	<b>16</b>
<b>4.0 PROTOCOL DEVIATIONS .....</b>	<b>17</b>
<b>5.0 CONCLUSIONS .....</b>	<b>18</b>
<b>5.1 Field Sub-Report Conclusion .....</b>	<b>18</b>
<b>5.2 Eurofins Analytical Sub-Report Conclusion .....</b>	<b>19</b>
<b>5.3 Monsanto Analytical Sub-Report Conclusion .....</b>	<b>19</b>
<b>5.4 Volatile Flux Estimation Sub-Report Conclusion .....</b>	<b>19</b>
<b>5.5 Indirect Flux and Off-Target Air Concentration and Vapor Deposition Modeling Sub-Report Conclusion .....</b>	<b>20</b>
<b>5.6 Spray Drift Deposition Modeling Sub-Report Conclusion .....</b>	<b>20</b>
<b>5.7 Plant Effects (Drift and Volatility) Sub-Report Conclusion .....</b>	<b>21</b>
<b>5.8 Study Conclusions .....</b>	<b>21</b>
<b>6.0 REFERENCES .....</b>	<b>23</b>
<b>7.0 APPENDICES SECTION .....</b>	<b>24</b>
Appendix A. Study Protocol and Study Protocol Changes .....	25

Appendix B. Lange Research and Consulting, Inc. Field Sub-Report .....	80
Appendix C. Eurofins EAG Agrosience, LLC Analytical Sub-Report .....	153
Appendix D. Monsanto Company Analytical Sub-Report .....	316
Appendix E. Monsanto Company Volatile Flux Estimation Using Aerodynamic (AD) and Integrated Horizontal Flux (IHF) Methods Sub-Report .....	468
Appendix F. Exponent, Inc. Indirect Flux and Off Target Modeling Sub-Report.....	487
Appendix G. Monsanto Company Spray Drift Deposition Modeling Sub-Report.....	545
Appendix H. Monsanto Company Plant Effects (Drift and Volatility) Sub-Report.....	561

## ABBREVIATIONS AND SYMBOLS

Abbreviation	Definition
A	acre
AD	Aerodynamic
a.e.	acid equivalent
°C	degrees Celsius
DAT	Days after treatment
EOF	Edge of field
°F	degrees Fahrenheit
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
GLP	Good Laboratory Practice
GPA	gallons per acre
ha	hectare(s)
IHF	Integrated Horizontal Flux
in	inch(es)
L	liter(s)
LRC	Lange Research and Consulting, Inc.
lb	pound(s)
LC-MS/MS	Liquid Chromatography Tandem Mass Spectrometry
LOQ	Limit of Quantitation
m	meter(s)
mm	millimeter(s)
Monsanto	Monsanto Company
mph	miles per hour
ng	nanogram(s)
NOAEC	no observed adverse effect concentration
NOAER	no observed adverse effect rate
NRCS	Natural Resources Conservation Service
PUF	polyurethane foam
QA	Quality Assurance
s	second(s)
SOP	Standard Operating Procedure
US EPA	United States Environmental Protection Agency
USDA	United States Department of Agriculture
µg	microgram(s)
UV	ultraviolet



## 1.0 INTRODUCTION

A field study measuring volatilization, spray drift, and plant effects of an XtendiMax tank mix was performed in July 2019 in Clinton County, Illinois. A tank mix of XtendiMax (MON 76980) + PowerMax (MON 79789) + Intact™ + MON 51817 was applied to a dicamba-tolerant soybean field that was surrounded by non-dicamba tolerant soybean. The purpose of this study was to evaluate potential for off-target dicamba volatilization and spray drift deposition, as well as potential plant effects from both sources.

This report is a summary of seven sub-reports that collectively present the results and conclusions from this study designed to evaluate the potential off-target movement of the dicamba tank mix application conducted in Clinton County, Illinois in July 2019. The following table summarizes important study information, dates, times, and data.

Parameter		Sub-Report (PI Company)
Study location	Shattuc, Clinton County, IL	Field (LRC)
Application acreage	21 acres	Field (LRC)
Application time	07/02/2019, 09:35 – 07/02/2019, 09:49 (~14 mins)	Field (LRC)
Test substance and tank mix partners	MON 76980 + MON 79789 + Intact™ + MON 51817	Field (LRC)
Vapor phase sampling duration (volatilization)	~7 days	Field (LRC)
Spray drift deposition sampling duration	~7 days	Field (LRC)
Max air temperature for duration of vapor phase sampling at 1.7 m from Main Met station	92.80° F, 33.78 C	Field (LRC)
Max air temperature during periods 1-3 of vapor phase sampling at 1.7m height from Main Met station	92.01 °F, 33.8 °C (Period 1)	Field (LRC)
Max surface (1 mm) soil temp for duration of vapor phase sampling from main met station	102 °F, 38.89 °C	Field (LRC)
Relative humidity range for duration of vapor phase sampling at 1.7 m from Main Met station	46% to 100%	Field (LRC)
Total precipitation during vapor phase sampling	2.35 in (59.69 mm)	Field (LRC)
Acceptable wind speed range during application	3-10 mi./hr., 1.34-4.47 meter/sec.	Field (LRC)
Average wind speed during application	4.881 mi./hr., 2.182 meter/sec.	Field (LRC)
Target wind direction for application	225°	Field (LRC)
Wind direction during application (vector average)	235.1°	Field (LRC)
Max relative humidity during application	75.39%	Field (LRC)
Soil type	Silt Loam	Field (LRC)
Soil pH	6.8	Field (LRC)

Parameter		Sub-Report (PI Company)
Source water pH	7.8	Field (LRC)
Tank mix pH	5.2	Analytical (Eurofins)
Target application rate	15 GPA	Field (LRC)
Actual application rate	15.4 GPA (15 GPA x 102.7%)	Field (LRC)
AD max flux and period	0.6790 ng/m <sup>2</sup> /sec, Period 1 (0-6 hours)	Flux (Monsanto)
IHF max flux and period	0.1392 ng/m <sup>2</sup> /sec, Period 1 (0-6 hours)	Flux (Monsanto)
Indirect max flux and period	0.30 ng/m <sup>2</sup> /sec, Period 1 (0-6 hours)	Off-target (Exponent)
Conservative estimate of total cumulative mass loss (% applied)	0.046% (Indirect method)	Off-target (Exponent)
Max of 90th percentile 24-hour total deposition at 5 m from EOF	7.48E-01 µg/m <sup>2</sup> (Dry Deposition)	Off-target (Exponent)
Max of 24-hour air concentration at 5 m from EOF	1.47 µg/m <sup>2</sup>	Off-target (Exponent)
Max buffer distance (transect location, period) (NOAER = 0.000522 application fraction)	18.08 m, DWB, Period 1 (1 hour)	Spray Drift (Monsanto)
Rainfall during 28-day plant effects sampling phase from the long duration meteorological station	3.5in. (90mm)	Plant Effects (Monsanto)
Application monitoring (tank mix % of target)	95%	Analytical (Eurofins)

## 2.0 STUDY OBJECTIVE

The purpose of this study is to measure dicamba spray drift deposition during application, dicamba volatility and deposition after the application, and quantify differences in plant effects via exposure of spray drift deposition and volatility exposure to non-tolerant soybeans, a surrogate for other non-target plant species. For detailed information regarding the study, refer to the study protocol and protocol changes in Appendix A.

The objective of this study was to determine off-target movement due to volatility and spray drift and resulting impacts to non-target plants (via volatility and drift) of a Monsanto dicamba herbicide formulation. The test substance was applied to a field planted with dicamba- and glyphosate-tolerant soybean (Asgrow AG41X8). The surrounding soybeans were planted with non-dicamba tolerant and, at a minimum, glyphosate-tolerant soybean of similar variety/maturity group (Dyna-Gro S40GL59). Use of non-dicamba tolerant soybeans in the surrounding application area allowed for dicamba exposure assessments. Air, spray deposition, and plant effects sampling data as well as meteorological data were collected and analyzed to estimate potential gaseous vapors that could result from volatilization and deposition over time following test substance applications. These data were then used to facilitate the development of dissipation curves, Indirect Flux modeling results, Integrated Horizontal Flux modeling results, Aerodynamic Flux modeling results, AERMOD deposition modeling results, and PERFUM air concentration modeling results. Visual plant effects ratings and height measurement data were used to quantify non-tolerant soybean exposure to volatility and spray drift.

This report describes the field, analytical, modeling, and statistical plant effects phases of the study concerning the test substance containing dicamba.

### **3.0 STUDY SUB-REPORTS**

#### **3.1 Field Sub-Report**

The Field Sub-Report was written by Lange Research and Consulting, Inc. and can be found in Appendix B. The sub-report includes Quality Assurance (QA) and Good Laboratory Practice (GLP) Compliance Statements. Information on the field phase of the study including: descriptions of plot layout, source water and soil characterization, tank mix preparation, deposition sample collection (spray drift), air sample collection (volatilization), sample controls (volatilization and spray drift deposition), and meteorological data collection are included in the Field Sub-Report.

#### **3.2 Analytical Sub-Reports**

An Analytical Sub-Report was written by Eurofins EAG Agrosience LLC and can be found in Appendix C. This sub-report, which includes a QA statement and GLP Compliance Statement, and was prepared by the Analytical Principal Investigator. Analytical results for pre-application, application, post-application, spray area, tank mix, and quality control samples are provided in the Analytical Sub-Report.

Eurofins performed LC-MS/MS sample analysis for PUF collectors (pre-application, post application, field spikes, and transit stability) using analytical method ME-2242, “LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps” (ME-2242-01). See Appendix D in the Analytical Sub-Report, Appendix C, for more details.

Eurofins performed HPLC-UV analysis for application monitoring filter papers using analytical method ME-2166, “Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification” (ME 2166-01). See Appendix B in the Analytical Sub-Report, Appendix C, for more details.

Eurofins performed HPLC-UV sample analysis for tank mix samples using analytical method ME-2154, “Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification” (ME-2154-01). See Appendix C in the Analytical Sub-Report, Appendix C, for more details.

An Analytical Sub-Report was written by Monsanto Company who performed LC-MS/MS sample analysis for spray drift deposition filter papers (upwind, downwind, left wind, right wind, and transit stability) using analytical method ME-1871, “Determination of Dicamba After Deposition on Filter Paper by LC-MS/MS (ME-1871-01). See Appendix B in the Analytical

Sub-Report, Appendix D, for more details. The sub-report does not include a QA statement as this is documented in the QA statement of the main report found above. The sub-report, which does include a GLP Compliance Statement, was prepared by the Analytical Principal Investigator and can be found in Appendix D.

### **3.3 Volatile Flux Estimation Sub-Report**

The Volatile Flux Estimation Using Aerodynamic (AD) and Integrated Horizontal Flux (IHF) Methods Sub-Report was written by Monsanto Company. The sub-report, which includes a QA statement and GLP Compliance Statement, was prepared by the Modeling Principal Investigator. The sub-report describing the results of the AD and IHF and Information on the modeling phase of the study including dicamba concentrations, outlier analysis, flux estimation methods (Integrated Horizontal and Aerodynamic Flux), and mass loss calculations are included in the Volatile Flux Estimation Sub-Report (Appendix E).

### **3.4 Indirect Flux and Off-Target Modeling Sub-Report**

The Indirect Flux and Off-Target Modeling Sub-Report builds from the Volatile Flux Estimation Sub-Report and was written by Exponent, Inc. The Indirect Flux and Off-Target Modeling Sub-Report does not include QA and GLP Compliance Statements, as this portion was not conducted per GLPs. This sub-report describes the results of the Indirect flux model and provides information on the PERFUM and AERMOD simulations from Lubbock, TX, Peoria, IL, and Raleigh, NC, which is included in the Indirect Flux and Off-Target Modeling Sub-Report (Appendix F).

### **3.5 Spray Drift Deposition Modeling Sub-Report**

The Spray Drift Deposition Modeling Sub-Report was written by Monsanto Company and was prepared by the Statistical Analysis Principal Investigator. The sub-report does not include a QA and GLP Compliance Statements as this is documented in the statements of the main report found above. Information on the results and statistical analysis (fraction of applied and buffer distances) are included in the Spray Drift Deposition Modeling Sub-Report (Appendix G).

### **3.6 Plant Effects (Drift and Volatilization) Sub-Report**

The Plant Effects (Drift and Volatilization) Sub-Report was written by Monsanto Company and was prepared by the Plant Effects Principal Investigator. The sub-report includes a GLP Compliance Statement and does not include a QA statement as this is documented in the QA statement of the main report found above. Information on the volatilization effects on plants and spray drift effects on plants are included in the Plant Effects Sub-Report, found in Appendix H.

## 4.0 PROTOCOL DEVIATIONS

Described below are the eight protocol deviations that occurred during the study. The deviations were determined to have minimal impact on the integrity of the study.

Protocol Change Document 4 dated February 25, 2020 contains five deviations to the protocol (Appendix A) and are described below.

- The soil characterization sample was shipped chilled instead of ambient. This deviation is not expected to have a negative effect on the study as the sample is assumed to be at least as stable under chilled conditions.
- Water holding capacity for 15 bar (disturbed) was not requested or performed for soil characterization. This deviation is not expected to have a negative effect on the study as these data were not intended for additional analysis within this study.
- The incorrect test was requested and performed for water characterization and the following parameters were not measured: potassium, carbonate, bicarbonate, nitrate, sulfate, chloride, and alkalinity. This deviation is not expected to have a negative effect on the study as these data were not intended for additional analysis within this study.
- The tractor sprayer speed was not calibrated 3 times prior to application. This deviation is not expected to have a negative effect on the study as the spray swaths were within an average of 3% of target pass times.
- A total of 47 deposition filter paper samples were unable to be collected from the 96, 120, and 168 hour collection periods due to rainfall. Samples from earlier periods of collection, where higher concentrations are expected to occur, resulted in levels of detection that were already too low to be used for deposition modeling. It is expected that these later uncollected samples would also contain levels of residue too low for modeling, and therefore this deviation is not expected to have a negative effect on the study.

Protocol Change Document 5 dated March 18, 2020 contains one deviation to the protocol (Appendix A) and is described below.

- The temperature of the test substance was not recorded during temporary storage from June 19, 2019 to June 30, 2019, prior to test substance application. Due to tank mix verification samples and application verification filter paper samples showing dicamba concentrations within the expected range, this deviation is not expected to have a negative effect on the study.

The following two deviations occurred during the analytical phase of the study.

- A Eurofins method deviation dated September 04, 2019 documented one instance during the study on which the percent relative standard deviation (RSD) of LOQ quality control (QC) fortification recoveries within an analytical set following analytical method ME-

2242 was >20% RSD. This is deemed to have no impact on the integrity of the study as the mean recovery is within the acceptable range of the method, and the overall RSD of LOQ fortifications in this portion of the study was 12.1%.

- A Eurofins SOP deviation dated August 27, 2019 documented one instance during the study on which the display settings for the pipette used to prepare the transit stability and field QC samples was not documented. This deviation was determined not to have had a significant negative impact on the integrity of the study as documentation supports that proper procedure and equipment were used, the procedure was witnessed by a second party, and the results of the fortifications samples were as expected.

## **5.0 CONCLUSIONS**

A study to assess potential off-target movement and non-target plant effects of dicamba was performed in July 2019 in Clinton County, Illinois. The study was designed to measure spray drift and volatility of dicamba following a spray application of MON 76980 (0.5 lb a.e./A) + MON 79789 (1.125 lb a.e./A) + Intact™ (0.5% v/v) + MON 51817 (1% v/v) to a plot of dicamba-tolerant soybeans with a surrounding area planted with non-dicamba tolerant soybeans. Measurements and quantification of spray drift deposition (during application) and deposition from volatilized dicamba (post-application), volatilization (post field application), and non-tolerant soybean plant effects via spray drift deposition and volatility exposure were assessed. Based on nozzle and sprayer verification, data recorded by the sprayer during application, tank mix samples, and application monitoring samples, the dicamba formulation was successfully applied near the target rate of 0.5 lb a.e./A.

### **5.1 Field Sub-Report Conclusion**

A field study measuring volatilization, spray drift, and plant effects was performed near Shattuc, Clinton County, Illinois to measure flux of dicamba following spray applications of a mixture of MON 76980 at a rate of 0.50 lb dicamba a.e./A (0.56 kg dicamba a.e./ha) to a field planted with both dicamba-tolerant and dicamba non-tolerant emergent soybean. Based on the pass time and spray calibration results, and the application verification samples, the dicamba formulation was successfully applied near the target rate of 0.5 lb a.e./A, with individual spray swaths ranging from 97.7 % to 106.9 % of the target rate. It is also presumed that the use of variable rate technology was able to adjust the total application rate closer to the target rate than what could be achieved based on calculated pass times and calibrated spray rate alone.

During the seven-day post-application sample phase of the study, based on minute-averaged data from the 10-meter main meteorological station, the maximum measured air temperature at 1.7 m height was 92.80 °F (33.78 °C) for the test plot. The maximum measured surface soil temperature (1 mm depth) was 102 °F (38.89 °C) for the test plot.

## 5.2 Eurofins Analytical Sub-Report Conclusion

Dicamba on application monitoring filter paper samples was analyzed using the current version of analytical method ME-2166. Method performance assessments within the study showed acceptable accuracy (average within 90-110%) and precision ( $\leq 5\%$  RSD) for application monitoring filter paper analyses.

Dicamba in tank mix samples was analyzed using the current version of analytical method ME-2154 with Eurofins modifications. Method performance assessments within the study showed acceptable accuracy (average within 90-110%) and precision ( $\leq 5\%$  RSD) for tank mix analyses.

Dicamba on PUF collectors was analyzed using the current version of analytical method ME-2242. In this study, the working range of the analytical method was 0.030 to 7.5 ng/PUF with a Limit of Quantitation (LOQ) of 0.1 ng/PUF. Method performance assessments within the study showed acceptable accuracy (average within 70-120%), precision ( $\leq 20\%$  RSD), and selectivity ( $\leq 30\%$  of the LOQ) for PUF collectors fortified with dicamba, with the exception of analytical Set 001 in which the LOQ fortifications resulted in an RSD of 20.7%.

## 5.3 Monsanto Analytical Sub-Report Conclusion

Dicamba on spray drift deposition filter paper collectors was analyzed using the current version of analytical method ME-1871. In this study, the working range of the analytical method was 0.0015 to 6.0  $\mu\text{g}/\text{filter}$  with an LOQ of 0.005  $\mu\text{g}/\text{filter}$ . Method performance assessments within the study showed acceptable accuracy (within 70-120%), precision ( $\leq 20\%$  RSD), and selectivity ( $\leq 30\%$  of the LOQ) for filter paper collectors fortified with dicamba. The calibration curves used a liner regression forced through zero model with 1/x weighting for quantitation, instead of a quadratic regression model with 1/x weighting.

## 5.4 Volatile Flux Estimation Sub-Report Conclusion

Volatile flux was estimated using IHF and AD methods following application of a tank mix containing MON 76980, MON 79789, Intact and MON 51817 on an 8.7 ha (296 m x 293 m) soybean field. Approximately 2.35" of rain was measured on the field during the 7-day sampling period after application. During the study, based on period-average data, the maximum temperature was 31.8°C (89.2°F) which occurred during period 4 (24-36 hours) at 48 cm height.

The flux profile (i.e. flux vs. time) had a similar range across the two methods except period 1. Highest flux was estimated was 0.6790 ng/m<sup>2</sup>/sec using AD method. The cumulative dicamba mass lost (as a percent of applied) during the study was 0.015%, and 0.036% based on IHF, and AD methods, respectively.

## **5.5 Indirect Flux and Off-Target Air Concentration and Vapor Deposition Modeling Sub-Report Conclusion**

The Indirect Method was used to determine fluxes during the hours following dicamba application on a field in Illinois. The 1-minute average measurements of meteorological parameters were further averaged to 1-hour blocks and prepared for air dispersion modeling. The period average modeled concentrations obtained with unit emission rate were linearly regressed with measured concentrations for the 8 sample points. The resulting slope of the regression passing through zero was used as an estimate of the flux from the field.

A comparison was made between measured and modeled concentrations using the indirect flux estimate at each monitor location for each of the measurement periods. Nighttime periods result in lower flux rates and light variable winds may contribute to more spatial variability between the modeled and observed results.

The peak calculated flux rate was 0.30 ng/m<sup>2</sup>/s, estimated during the first period. Based on the indirect flux profile, it was estimated that the cumulative dicamba (acid equivalent) mass lost from the field over the entire 166-hour duration of the study was 0.05% of applied.

The deposition estimates were made using AERMOD, combined with flux data developed by Monsanto and Exponent and meteorological data from the National Weather Service. The concentration estimates were made using PERFUM (version 2.5), combined with flux data developed by Monsanto and Exponent and meteorological data provided by the U.S. EPA.

At 5 meters from the edge of the field, the maximum 24-hour average dry deposition ranged from  $8.6 \times 10^{-7}$  to  $1.5 \times 10^{-6}$  g/m<sup>2</sup> and the maximum wet deposition ranged from  $7.3 \times 10^{-8}$  to  $1.4 \times 10^{-7}$  g/m<sup>2</sup>. At the same distance, 5 meters from the edge of the field, the 90th percentile dry deposition ranged from  $4.7 \times 10^{-7}$  to  $7.5 \times 10^{-7}$  g/m<sup>2</sup>, and the 90th percentile wet deposition ranged from  $1.6 \times 10^{-9}$  to  $5.3 \times 10^{-9}$  g/m<sup>2</sup>. The highest depositions were estimated using meteorological data from Raleigh, North Carolina and Peoria, Illinois. Generally, the 90th percentile values are about 42-56% of the maximum values for dry deposition, and 1-6% of the maximum values for wet deposition.

The estimated 24-hour average air concentrations at 5 m (16.4 ft) from the edge of the field ranged from 1.9 to 2.8 ng/m<sup>3</sup>. Similarly, the 24-hour air concentrations at 10 m (32.8 ft) ranged from 1.8 to 2.6 ng/m<sup>3</sup>, 25 m (82 ft) ranged from 1.6 to 2.3 ng/m<sup>3</sup> and 50 m (164 ft) ranged from 1.3 to 1.9 ng/m<sup>3</sup>.

## **5.6 Spray Drift Deposition Modeling Sub-Report Conclusion**

Deposition of dicamba above the NOAER (0.000261 lbs a.e./A, equivalent to an application fraction of 0.000522 for an application rate of 0.5 lbs a.e./A) was detected in all transects of the downwind and left-wind sides of the application area (DWA, DWB, DWC, LWA, and LWB) in period 1 (1-hour). Estimated distances, to reach the NOAER for period 1 datasets ranged from



<10 m (32.8 ft) to 18.08 m (59.3 ft) in the downwind direction and from 12.66 m (41.5 ft) to 16.52 m (54.2 ft) in the left-wind direction. All other transects and periods were excluded from statistical analysis due to one or several of the following reasons: (1) Zero or small number of observations, (2) Lack of trend in deposition values, or (3) Deposition values being much less than the no observed adverse effect rate.

### **5.7 Plant Effects (Drift and Volatility) Sub-Report Conclusion**

Off-target movement of dicamba due to volatility and spray drift following a large acre application to DT soybeans surrounded by non-tolerant soybeans was assessed by comparing plant heights and visual plant response along transects oriented perpendicular to the sprayed field edge and radially from the plot corners. Soybeans without the dicamba tolerance trait are known to be sensitive to dicamba and were used as a bioindicator of effects on plants. Plant effects were measured at 0, 14, and 28 days after treatment (DAT±2) along each drift and volatility transect.

Overall, the soybeans in the volatility transects exhibited only minor visual symptomology and no effects on plant height due to volatility were observed. Plant effects in the transects located downwind of the predominant winds at application were more pronounced (downwind, left wind, and NW corner). The no-effect distance for plant height in these transects ranged from 10 to 23.5 m (33 to 77 ft).

### **5.8 Study Conclusions**

Based on the verification of the nozzles and sprayer, sprayer data, and the application monitoring samples, the dicamba formulation was applied near the target rate of 0.5 lb a.e./A. During the application, average wind speed was in the target range of 3 – 10 mph and wind direction was within ±30° of the target direction.

Soybeans without the dicamba tolerance trait are known to be highly sensitive to dicamba and were used as a bioindicator of non-target plant effects. The soybeans in the volatility transects (transects that were tarped during application to limit exposure to spray drift) exhibited only minor (≤10%) visual symptomology and no effects on plant height were observed. This is consistent with the volatile flux estimates generated from the field-collected data, and the modeled off-target air concentration and vapor deposition, as described in the following paragraphs.

Volatile flux was estimated using three methods: Indirect, IHF, and AD methods. The method resulting in the highest total estimated mass lost was the Indirect method, which estimated that 0.05% of applied dicamba was lost during the seven-day sample collection period of the study. The highest estimated flux came from the AD method during period 1 and was 0.67902 ng/m<sup>2</sup>/s.

Off-target air concentrations were estimated using PERFUM, using a conservatively estimated 24-hour flux profile and meteorological inputs for three representative locations. The maximum

predicted 95th percentile 24-hour average air concentration of dicamba was 2.8 ng/m<sup>3</sup>, significantly less than the soybean no-observed-adverse-effect-concentration (per MRID 50578901 [Gavlick, 2016]: NOAEC 138 ng/m<sup>3</sup>).

Off-target deposition was estimated using AERMOD, using a conservatively estimated 24-hour flux profile and meteorological inputs for three representative locations. The maximum predicted 90th percentile 24-hour total deposition across the three locations (5 m from the edge of the treated field) was 7.48E-01 µg/m<sup>2</sup>, as dry deposition, significantly less than the vegetative vigor no observed effect rate (per EPA DER of MRID 47815102 [US EPA, 2013]: NOER = 2.61 X 10<sup>-4</sup> lb/A = 29.1 µg/m<sup>2</sup>).

Deposition of dicamba above the NOAER (0.000261 lbs a.e./A, equivalent to an application fraction of 0.000522 for an application rate of 0.5 lbs a.e./A) was detected in all transects of the downwind and left wind directions in the 1-hour period), the period representing application and immediately post-application. Estimated distances from edge of field to reach the NOAER for soybean in period 1 ranged from <10 m (32.8 ft) to 18.08 m (59.06 ft) and from 12.66 m (41.5 ft) and 16.52 m (54.2 ft) in the downwind and left wind directions, respectively.

The no-effect distance for plant height in the downwind drift (un-tarped) transects (downwind A and C, northwest diagonal, and left-wind A and B) ranged from 32.81 ft (10 m) to 77 ft (23.47 m.) No-effect distances were 0 ft in all other directions for drift transects (un-tarped) and all directions for volatility (tarped) transects, indicating the primary route of off-target movement was from spray drift in the downwind direction at the time of spray application. These results are consistent with the filter paper sample analytical results and off-target spray drift deposition modeling, as described in previous paragraph.

## 6.0 REFERENCES

US EPA. (1998). *Spray Drift Test Guidelines, OPPTS 840.1200 Spray Drift Field Deposition*. United States Environmental Protection Agency, Prevention, Pesticides, and Toxic Substances. EPA 712-C-98-112.

US EPA. (2008b). *Fate, Transport and Transformation Test Guidelines, OPPTS 835.8100 Field Volatility*. United States Environmental Protection Agency, Prevention, Pesticides, and Toxic Substances. EPA 712-C-08-024.

## **7.0 APPENDICES SECTION**

## **Appendix A. Study Protocol and Study Protocol Changes**

**STUDY PROTOCOL**

Study Number	REG-2019-0035
Study Title	Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois
Sponsor	Monsanto Company
Sponsor Representative	Thomas B. Orr, M.S. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9347 E-mail: <a href="mailto:thomas.b.orr@monsanto.com">thomas.b.orr@monsanto.com</a>
Study Director	Will Griese Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9341 E-mail: <a href="mailto:will.j.griese@monsanto.com">will.j.griese@monsanto.com</a>
Primary Testing Facility & Management	Rodrigo Sala, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-6483 E-mail: <a href="mailto:rodrigo.sala@monsanto.com">rodrigo.sala@monsanto.com</a>
Testing Facility – Analytical Chemistry Principal Investigator	Matthew Rebstock Eurofins EAG Agrosience, LLC 7200 East ABC Lane Columbia, MO 65202 Phone: +1-573-777-6385 E-mail: <a href="mailto:MatthewRebstock@eurofinsus.com">MatthewRebstock@eurofinsus.com</a>
Testing Facility – Analytical Chemistry Principal Investigator	Michael R. Shepard, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1- 636-737-9332 E-mail: <a href="mailto:michael.r.shepard.jr@monsanto.com">michael.r.shepard.jr@monsanto.com</a>
Testing Facility – Soil and Water Characterization Principal Investigator	Larry Wikoff AGVISE Laboratories 604 Highway 15 West P.O. Box 510 Northwood, ND 58267 Phone: +1-701-587-6010 E-mail: <a href="mailto:lwikoff@polarcomm.com">lwikoff@polarcomm.com</a>

Test Site	Chris Schaubert Schaubert Farms Shattuc, IL 62231
Field Research Principal Investigator – Volatility and Deposition	Alex Gibbs Lange Research and Consulting, Inc. 4746 W. Jennifer Ave. Suite 105 Fresno, CA 93722 Phone: +1-537-355-8608 E-mail: <a href="mailto:alex@langerc.com">alex@langerc.com</a>
Modelling (Statistical Analyses) Principal Investigator – Volatility	Richard Reiss, Sc.D. Exponent, Inc. 1800 Diagonal Road, Suite 500 Alexandria, VA 22314 Phone: +1-571-227-7228 E-mail: <a href="mailto:rreiss@exponent.com">rreiss@exponent.com</a>
Modelling (Statistical Analyses) Principal Investigator – Volatility	Naresh Pai, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9343 E-mail: <a href="mailto:naresh.pai@monsanto.com">naresh.pai@monsanto.com</a>
Field Research Principal Investigator – Plant Effects	Timothy Fredricks, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9307 E-mail: <a href="mailto:timothy.b.fredricks@monsanto.com">timothy.b.fredricks@monsanto.com</a>
Statistical Analyses Principal Investigator – Deposition and Plant Effects	Adam Schapaugh, Ph.D. Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9362 E-mail: <a href="mailto:adam.schapaugh@monsanto.com">adam.schapaugh@monsanto.com</a>
Primary Quality Assurance Unit	Lori Rodaway Monsanto Company 700 Chesterfield Parkway West Chesterfield, MO 63017 Phone: +1-636-737-9405 E-mail: <a href="mailto:lori.rodaway@monsanto.com">lori.rodaway@monsanto.com</a>

Approved by:



Thomas B. Orr, M.S.  
Sponsor Representative  
Monsanto Company

5/28/2019

Date



Will Gries  
Study Director  
Monsanto Company

5/31/2019

Date

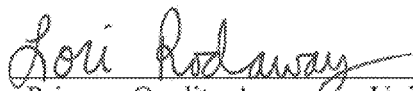


Rodrigo Sala, Ph.D.  
Primary Testing Facility & Management  
Monsanto Company

5/31/19

Date

Reviewed by:



Lori Rodaway  
Primary Quality Assurance Unit  
Monsanto Company

29 May 19

Date



Accepted by:

  
Analytical Chemistry Principal Investigator  
Eurofins EAG Agrosience, LLC24 May 2019  
DateAnalytical Chemistry Principal Investigator  
Monsanto Company

Date

Soil and Water Characterization Principal Investigator  
AGVISE Laboratories

Date

Field Research Principal Investigator – Volatility and Deposition  
Lange Research and Consulting

Date

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Exponent, Inc.

Date

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Monsanto Company

Date

Field Research Principal Investigator – Plant Effects  
Monsanto Company

Date

Statistical Analyses Principal Investigator – Deposition and Plant Effects  
Monsanto Company

Date

Accepted by:

---

Analytical Chemistry Principal Investigator  
Eurofins EAG Agroscience, LLC

---

Date

---

  
Analytical Chemistry Principal Investigator  
Monsanto Company

---

5/28/2019  
Date

---

Soil and Water Characterization Principal Investigator  
AGVISE Laboratories

---

Date

---

Field Research Principal Investigator – Volatility and Deposition  
Lange Research and Consulting

---

Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Exponent, Inc.

---

Date

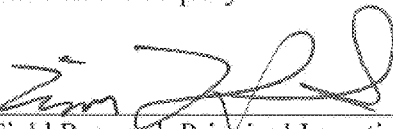
---

Naveen Sai  
Modelling (Statistical Analyses) Principal Investigator – Volatility  
Monsanto Company

---

5/23/2019  
Date


---

  
Field Research Principal Investigator – Plant Effects  
Monsanto Company

---

05/28/2019  
Date

---

  
Statistical Analyses Principal Investigator – Deposition and Plant Effects  
Monsanto Company

---

5/29/2019  
Date

Monsanto Company Confidential

Accepted by:

---

Analytical Chemistry Principal Investigator  
Eurofins EAG Agrosience, LLC


---

Date

---

Analytical Chemistry Principal Investigator  
Monsanto Company

---

Date

---

Soil and Water Characterization Principal Investigator  
AGVISE Laboratories

---

Date

5/23/19

---

Field Research Principal Investigator – Volatility and Deposition  
Lange Research and Consulting

---

Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Exponent, Inc.

---

Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Monsanto Company

---

Date

---

Field Research Principal Investigator – Plant Effects  
Monsanto Company

---

Date

---

Statistical Analyses Principal Investigator – Deposition and Plant Effects  
Monsanto Company

---

Date*Monsanto Company Confidential*

Accepted by:

---

Analytical Chemistry Principal Investigator  
Eurofins EAG Agrosience, LLC

---

Date

---

Analytical Chemistry Principal Investigator  
Monsanto Company

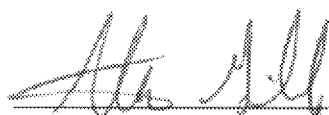
---

Date

---

Soil and Water Characterization Principal Investigator  
AGVISE Laboratories

---

Date

---

Field Research Principal Investigator – Volatility and Deposition  
Lange Research and Consulting

---

5/24/2019  
Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Exponent, Inc.

---

Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Monsanto Company

---

Date

---

Field Research Principal Investigator – Plant Effects  
Monsanto Company

---

Date

---

Statistical Analyses Principal Investigator – Deposition and Plant Effects  
Monsanto Company

---

Date*Monsanto Company Confidential*

Accepted by:

---

Analytical Chemistry Principal Investigator  
Eurofins EAG Agrosience, LLC

---

Date

---

Analytical Chemistry Principal Investigator  
Monsanto Company

---

Date

---

Soil and Water Characterization Principal Investigator  
AGVISE Laboratories

---

Date

---

Field Research Principal Investigator – Volatility and Deposition  
Lange Research and Consulting

---

Date

5/23/17

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Exponent, Inc.

---

Date

---

Modelling (Statistical Analyses) Principal Investigator – Volatility  
Monsanto Company

---

Date

---

Field Research Principal Investigator – Plant Effects  
Monsanto Company

---

Date

---

Statistical Analyses Principal Investigator – Deposition and Plant Effects  
Monsanto Company

---

Date

## 1.0 Regulatory Compliance

### 1.1 GLP Compliance

This study will be conducted in accordance with the United States EPA Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) Good Laboratory Practice (GLP) Standards (40 CFR 160). The indirect flux modeling as well as air dispersion modeling for off-target air concentration and vapor deposition will be conducted by Exponent, Inc. which will not be under GLP, however all quality measures taken will be documented in their sub-report.

### 1.2 Quality Assurance

Monsanto Company Quality Assurance Unit (QAU) will have the overall responsibility of providing QA oversight for this study.

Lange Research and Consulting QAU will conduct inspections/audits of critical events at intervals to ensure the integrity of the study for the field phase, including but not limited to: site preparation, application, a component of each measurement event type over the course of the study, field notebook, raw field data (volatility and deposition related), and review of the field sub-report.

AGVISE QAU will conduct inspections/audits of the soil and water characterization process and raw data.

Monsanto Company QAU will conduct inspections/audits of critical events at intervals to ensure the integrity of the study for the plant effects field notebook and raw field data, and review calculations performed by Monsanto Company staff (statistical analyses) and sub-reports/attachments (plant effects field phase, analytical phase and modeling phase).

Eurofins QAU will conduct inspections/audits of at least one critical event per high-concentration sample type (application monitoring or tank mix) and at least one critical event for PUF sample types (pre-application, post-application, field exposed spikes, or transit stability) for the analytical phase. Eurofins QAU will also review raw analytical data, calculations performed by staff (calibration curve and quantifying results related) and sub-report (analytical phase and results).

Monsanto Company QAU will conduct inspections/audits of at least one critical event for trace filter paper sample types (post-application or transit stability) for the analytical phase. Monsanto Company QAU will also review raw analytical data, calculations performed by staff (calibration curve and quantifying results related) and sub-report (analytical phase and results).

Monsanto Company QAU will conduct inspection/audits of dissipation and flux calculations (AD/IHF), statistical analyses, and data handling performed by Monsanto Company and sub-report(s).

Exponent will document their quality measures in their sub-report for indirect flux calculations as well the air dispersion modeling for off-target air concentration and vapor deposition.

The findings of these inspections/audits will be reported to the Study Director and Primary Testing Facility Management in a timely manner. Any problems which are likely to affect study integrity found during the course of an inspection/audit shall be brought to the attention of the Study Director and Primary Testing Facility Management immediately. Each QAU will provide a Quality Assurance Statement for their respective portions of the study within their respective reports, sub-reports, or attachments.

Any outsourcing of the QAU duties must be approved by the Study Director and Sponsor Representative prior to inspection/audit initiation.

### **1.3 Standards Cited**

The study design described in the protocol draws from the following methods: USEPA (revised 2016), ISO (2005), ASABE (revised 2013), USEPA Spray Drift Test Guideline, OPPTS 840.1200 Spray Drift Field Deposition (1998), USEPA Fate, Transport, and Transformation Test Guideline, OPPTS 835.8100: Field Volatility (2008), and a plant effects rating scale consistent with visual plant response ratings described in Frans and Talbert (1977).

### **1.4 Monsanto Company Global Stewardship and Compliance**

Label instructions will be adhered to for test substance and tank mix partners. Details of application will be documented in the field notebook.

Dicamba application records must be kept for each application and include detailed information regarding application conditions. Refer to label for complete record requirements. Use the dicamba application record keeping form available at <http://www.xtendimaxapplicationrequirements.com/Documents/XtendiMaxStewardshipRecordKeepingForm.pdf> or functional equivalent. These records must be generated on the same day, and as soon as practical after application.

### **1.5 Crop Destruct**

Crop destruct will not be required.

## **2.0 Purpose**

Monsanto Company has developed crops, including soybean and cotton, with a genetic trait that confers tolerance to dicamba herbicide. These herbicide-tolerant crops allow in-crop applications of dicamba herbicide for the control of broad-leaf weeds, including hard-to-control weeds. Monsanto Company has also developed dicamba formulations for use with dicamba-tolerant crops. Because dicamba-susceptible plant species may be adjacent to dicamba-tolerant crops where

dicamba formulations are applied, it is important to understand the potential for dicamba to move off-site via spray drift, deposition, and/or volatility. The purpose of this study is to measure spray drift during and deposition after field application, volatility after the application, and quantify differences in plant effects via exposure of spray drift/deposition and volatility to non-tolerant soybeans that serve as a surrogate for other non-target plant species.

### 3.0 Study Timeline

Proposed Experimental Start	Date: May 2019
Proposed Field Phase Termination	Date: July 2019
Proposed Experimental Termination	Date: August 2019
Proposed Study Completion	Date: September 2019

### 4.0 Test, Control, and Reference Substances

#### 4.1 Test Substance and Tank Mix Partners

The test substance and tank mix partners used in this study will be provided by the sponsor to the Lange Research and Consulting (LRC) facility in Moberly, MO, from where it will be transported to the test site. They will be prepared as a tank mixture at the test site and used in a single application to assess deposition of, volatility of, and plant effects to the primary active ingredient, dicamba.

Test Substance	
Identification	MON 76980
Active Ingredient	Diglycolamine salt of dicamba
Label a.e. Content (nominal)	29.0% (wt) Dicamba
Lot Number	To be documented in the field notebook

Tank Mix Partner 1	
Identification	MON 79789
Active Ingredient	Glyphosate potassium salt
Label a.e. Content (nominal)	39.8% (wt) glyphosate
Lot Number	To be documented in the field notebook

Tank Mix Partner 2	
Identification	Intact™
Principal Functioning Agents (PFA)	Polyethylene glycol, choline chloride, guar gum
Labeled PFA Content (nominal)	43.18%
Lot Number	To be documented in the field notebook



<b>Tank Mix Partner 3</b>	
Identification	MON 51817
Principal Functioning Agents (PFA)	Potassium acetate
Labeled PFA Content (nominal)	50%
Lot Number	To be documented in the field notebook

#### 4.2 Control Substance

There is no control substance in this study.

#### 4.3 Reference Substance

No reference substances will be applied in the field. Analytical reference standard used in this study will be provided by the Sponsor to the Testing Facilities overseen by the Analytical Chemistry Principal Investigators. Dicamba analytical reference standard will be used in the analytical phase of the study.

<b>Analytical Reference Substance</b>	
Identification	Dicamba
Active Ingredient	3,6-dichloro-2-methoxybenzoic acid
Purity	99.4%
Lot Number	To be documented in the analytical raw data

Other analytical standards used in the analytical phase of the study will be documented in the analytical raw data.

#### 4.4 Characterization of Test and Reference Substances

The concentrated test substance and the analytical reference standard(s) will be characterized by Monsanto Company. Certificates of analysis for the test substance and analytical reference standard(s) will be retained in the study file and reported.

#### 5.0 Test System and Justification of Test System

The study will be a test plot planted with both dicamba-tolerant and non-dicamba-tolerant soybeans and conducted at a test plot centered approximately (38.667412°, -89.152219°), near Shattuc, IL. The state of Illinois and Clinton County represent a key soybean growing and high dicamba use region.

The application area will be planted with soybeans tolerant to both dicamba and glyphosate. The surrounding soybeans will be planted with non-dicamba tolerant and, at a minimum, glyphosate-tolerant soybeans of a similar variety/maturity group as possible (variety/maturity to be

documented in the respective field notebooks). Use of non-dicamba tolerant soybeans in the area surrounding the application area allows for the assessment of dicamba exposure.

### **Application Monitoring**

Application monitoring of the tank mix applied to the test system will be conducted using in-swath filter paper collectors (Whatman #3, 12.5 cm diameter) placed within the application area (dicamba- and glyphosate-tolerant soybean area) and homogenized tank mix samples collected before and after application.

Collection and analysis of in-swath filter papers collected following application and homogenized tank mix samples collected before and after application will be used to assess that the target spray application of dicamba is similar to post-emergent, field applications of a commercial formulation containing dicamba.

### **Spray Drift**

Spray drift evaluations of the test system will be conducted using filter paper collectors (Whatman #1, 15 cm diameter) placed at specific distances in all directions from the edge of the application area (non-dicamba tolerant soybean area).

Collection and analysis of filter paper collectors following application of the homogenized tank mix will be used to quantify the deposition pattern of dicamba and potential to move off-target.

### **Field Volatility**

Field volatility evaluations of the test system will be conducted using polyurethane foam (PUF) collectors (e.g., SKC item number 226-92) placed at specific heights within a field planted with dicamba-tolerant soybean (center mast) and placed at specific locations and height within the surrounding field planted with non-dicamba tolerant soybeans (perimeter).

Collection and analysis of PUF collectors following application of the homogenized tank mix will be used to quantify field volatility of dicamba and potential to move off-target.

### **Plant Effects**

The test system will contain a field planted with dicamba-tolerant soybeans surrounded by non-dicamba tolerant soybeans.

Soybeans without the dicamba tolerance trait have been shown to be sensitive to dicamba (Porch 2009) and will be used as a bio-indicator of plant effects on sensitive species. Observations of plant visual symptomology and plant height measurements will be collected following application of the homogenized tank mix and will be used to determine plant response to dicamba that has potentially moved off-target.

## 5.1 Identification of Test System and Samples

Test plot identification will include staking out (or otherwise clearly identifying) the spray area, spray drift/deposition sample locations, air sampler locations, plant effects transect locations, and weather station locations with at least one stake identifying at a minimum the Sponsor protocol number (REG-2019-0035).

### Application Monitoring

Each in-swath filter paper collector set (4 individual samples) will be unique by its location in the field. The associated collection containers will be labeled to indicate the set location and individual sample identification code.

Each tank mix sample will be unique by its time period collected (pre-application and post-application). The associated collection containers will be labeled to indicate its time period collected (pre-application and post-application) and individual sample identification code.

### Spray Drift

Each filter paper collector will be unique by its location in the field and its time period. The associated collection containers will be labeled to indicate its location, time period, and individual sample identification code.

### Field Volatility

Each PUF collector and associated collection vials will be labeled to indicate its location, height, time period, and individual sample identification code.

### Plant Effects

A control plot will be clearly staked and uniquely identified upwind from the application area. The associated field notebook will be labeled to indicate location and time period of evaluation.

Each transect boundary will be clearly staked and uniquely identified by location and type (drift/volatility). Based on row spacing and orientation, distances from edge of field will be measured and marked  $\pm 1$  row/meter. The associated field notebook will be labeled to indicate location, transect and type, distance from edge of field, and time period of evaluation.

## 6.0 Experimental Design

A large-acre field study will be conducted to examine primary and secondary off-site movement of dicamba after application to post-emergent soybeans using combined methodologies of spray drift/deposition, field volatility, and plant effects. In summary, a single treatment of a homogenized tank mix spray solution containing dicamba will be applied to a cropped application area using ground boom application equipment. Data will be collected from within the cropped test plot and in surrounding directions with details described below.

## 6.1 Site Selection

The site will be an approximate 117-acre soybean field planted with both dicamba-tolerant and non-dicamba tolerant soybeans located at approximately (38.667412°, -89.152219°), near Shattuc, IL. The state of Illinois and Clinton County represent a key soybean growing and high dicamba use region.

The application area will be placed within the field to target having minimal wind obstructions (buildings, hedgerow, etc.) and target being located approximately 1,000 feet away from other anticipated dicamba applications, if possible, occurring up to one week before the application for this study and through four weeks after the application.

## 6.2 Field Preparation and Maintenance

Herbicides containing dicamba or other auxin-like herbicides may not be used up to 120 days prior to planting and throughout the study duration; exceptions must be coordinated with and documented by the Study Director or delegate. Other herbicides may be used for controlling any vegetation in the field prior to the start of the study. No additional applications may be made while data collection is in progress (i.e., 0-28 DAT); exceptions must be coordinated with and documented by the Study Director or delegate. Applicator will take care to not damage, physically or chemically, areas being utilized for plant effects ratings prior to the start of the study.

Prior to planting the field, the corners of the application area will be marked in order to maintain clear distinction between dicamba-tolerant and non-dicamba tolerant soybean areas.

## 6.3 Test Plot Diagram

A test plot diagram will be created using field notes, GPS coordinates of key locations and weather station data.

## 6.4 Test Plot Preparation

The application area will be approximately 293 m by 293 m (960 ft x 960 ft; ~21.2 acres) planted in dicamba- and glyphosate-tolerant soybeans located within an approximate 117-acre field. The surrounding acreage will be planted in non-dicamba tolerant and, at a minimum, glyphosate-tolerant soybean.

Based on the approximate 117-acre field size, there will be an approximate 300 ft minimum no-spray buffer around the application area. Incursions of other non-sensitive cover types, roads, or other structures into the 300 ft no-spray buffer are in general prohibited. Minor incursions may be acceptable but must be approved by Study Director.

Planting date, planting density, and row spacing of each soybean type will be documented in the associated field notebooks.

Every attempt will be made to plant the test plot so that two opposite edges are predominately oriented perpendicular to the estimated prevailing wind direction for the targeted application window (see Section 3.0 and Figure 1).

## 6.5 Test Substance Storage and Container Handling

The test substance and tank mix partners will be shipped to the Field PI's facility in Moberly, MO from Monsanto Company. The test substance and tank mix partners will be stored under label conditions in a monitored pesticide storage area adequate to preserve identity, purity, and stability of the test substance and tank mix partners. Chain of custody documents will be retained in the study file.

Test substance and tank mix partners will be shipped to:

Lange Research and Consulting  
Attn: Alex Gibbs  
1856 County Road 2630  
Moberly, MO 65270-5804

## 6.6 Test Substance Application

Target application of the homogenized tank mix will be representative of typical post-emergence soybean (vegetative stage; V2 – before R1).

The test substance and tank mix partners will be mixed with a water carrier and delivered at a target rate of at least 140.31 L/ha (15 gallons per acre). Spray pressures for the TTI 11004 nozzles will be targeted at 4.3 bar (63 psi), which are expected to achieve the desired application rate of 140.31 L/ha (15 gallons per acre) at a vehicle speed of approximately 16 km/h (10 mph). Actual pressures used during application may vary slightly depending on the sprayer type and if rate controller technology is available.

The target boom height for application will be approximately 51 cm (20 in) above the canopy. The sprayer will target to apply 0.5 lb dicamba a.e./A for a total application area of approximately 20 acres. The spray application volume will be verified through equipment calibration and data recorded by the sprayer. Applied spray application volume rates will be documented in the field notebook.

Application pattern (e.g., spray swaths) may differ from standards described in Section 1.3 to maintain the integrity of the buffer crop being evaluated for plant effects on all four sides of the application area. Designated ingress and egress areas for the sprayer will be pre-determined. Actual spray path will be documented in the field notebook.

The application will be a homogenized tank mix of the following formulations and rates:

MON 76980 (22 oz/A) + MON 79789 (32 oz/A) + Intact™ (0.5% v/v) + MON 51817 (1% v/v)

Rates were derived based on target post-emergence application rate (lb a.e./A) and nominal product loading information provided on the product labels as summarized below.

Test Substance	Target Application Rate (mass rate)		Nominal Product Loading				Target Application Rate (volume rate)	
MON 76980	0.5	lb a.e./A	2.9	lb a.e./gal	29.0	% w/w	22	oz/A
	560	g a.e./ha	350	g a.e./L			1.6	L/ha

Tank Mix Partners	Target Application Rate (mass rate)		Nominal Product Loading				Target Application Rate (volume rate)	
MON 79789	1.125	lb a.e./A	4.5	lb a.e./gal	39.8	% w/w	32	oz/A
	1261	g a.e./ha	540	g a.e./L			2.3	L/ha
Intact™	N/A	N/A	N/A		N/A		0.5	% v/v
MON 51817	N/A	N/A	N/A		N/A		1	% v/v

### Equipment Calibration

The sprayer will be calibrated by checking the flow rate through the sprayer prior to conducting the spray application. Sprayer output calibration will include measuring the volume output of water per unit time for each nozzle at a specified spray pressure. This process will be replicated three times for each nozzle (TTI 11004) included in the study. The average sprayer output must be within 5% of the target application volume.

The forward speed will be calibrated by timing the duration required, in seconds, to drive a known distance using markers – this process will be repeated three times. If necessary, the tractor speed will be adjusted based on the average speed recorded during equipment calibration to deliver at least 140.31 L/ha (15 gallons per acre) application volume. The target rate may vary based on the results of the equipment calibration.

### Preparation of Tank Mix

The amount of test substance to use in the tank mix will be based on the target rate of 0.5 lb dicamba a.e./A. The amount of tank mix prepared will be sufficient to ensure proper mixing, line charging, application and tank mix sampling. Tank mixing will be conducted according to test substance label instructions and will be documented in the field notebook.

A field pH of the source water prior to mixing and of the homogenized tank mix solution prior to application will be collected using a calibrated field pH meter and results recorded in the field notebook.

### Application Conditions

Spray application will be made during wind conditions of approximately 3 - 10 mph. However, actual wind speeds will depend on the weather conditions on the day of application. To minimize

off-target drift, the maximum sustained wind speed will not exceed 10 mph. To minimize the potential for atmospheric inversions, wind speeds will not be less than 3 mph at the start of the application.

Every attempt will be made to apply to the application area with wind direction perpendicular to two edges of the field (see Figure 1). Actual wind direction will depend on weather conditions on the day of application.

Application conditions will be recorded in the field notebook.

### **Application Timing**

Spray application timing will target starting in the morning between approximately one-hour after sunrise to noon local time. During the spray application, the clock time in Hours:Minutes will be recorded at the start of the application and the time in seconds will be recorded for time required to spray.

### **Spray Equipment Cleaning**

The spray equipment will be triple rinsed prior to the application and, per label, triple rinsed after the application. Details regarding the tank cleaning will be documented in the associated field notebook.

### **Safety Precautions**

The test substance and tank mix partners must be applied in accordance with the directions for MON 76980, MON 79789, Intact<sup>TM</sup>, and MON 51817 with an emphasis on any specified label requirements for protective clothing. Good agronomic safety practices will be followed regarding the use of long-sleeved shirt, long pants, waterproof gloves, and shoes plus socks. A SDS will be provided with each test substance and tank mix partner.

## **6.7 Sample Collection and Identification**

### **6.7.1 Soil and Source Water Characterization**

Soil from the test plot will be collected for characterization prior to the test substance application.

Fifteen cores will be extracted to a depth of 6 inches from across the test plot. The 15 cores will be combined, and the resulting composite will be thoroughly mixed before a sub-sample (minimum 500 g) will be removed for shipment to the soil testing laboratory. The container for the sub-sample of the disturbed soil will be labeled with the study number, site location, event description, date, and depth increment.

The sub-sample will be shipped at ambient temperature to the soil testing laboratory and analyzed to determine soil texture (percent sand, silt, and clay), percent organic matter, pH, cation exchange capacity, water holding capacity at 1/3 and 15 bar (disturbed), and bulk density (disturbed) (i.e., AGVISE Laboratories' Series II plus 15 bar water holding capacity test).

Ample amount of sprayer source water will be collected and shipped on ice to the water testing laboratory and analyzed to determine pH, calcium, magnesium, sodium, hardness, sodium adsorption ratio (SAR), conductivity, potassium, carbonate, bicarbonate, nitrate, sulfate, chloride, and alkalinity (i.e., AGVISE Laboratories' Series 5). The sprayer water will be collected from the water source prior to test system tank mixing. The container(s) for the sprayer water sample will be labeled with the study number, site location, event description, date, and time period collected.

Soil characterization and sprayer water analysis will be performed in compliance with GLP by AGVISE Laboratories, Inc., Northwood, North Dakota. Summary results will be included in a final report.

#### **6.7.2 Tank Mix Samples**

Tank mix samples will be collected by LRC staff and analyzed by Eurofins to verify the amount of dicamba present in the tank mix solution for two time points: 1) after the tank mix of the test substance has been fully homogenized prior to application (pre-application) and 2) following application (post-application). Three replicate samples of at least 50 mL each will be collected in a uniquely labeled container for a total of 6 tank mix samples.

Tank mix samples must be kept in separate storage areas and shipment containers (i.e., separate coolers) from all other samples during storage and shipping to avoid the potential for cross contamination. Tank mix samples will be stored and shipped under ambient conditions.

#### **6.7.3 In-Swath Samples**

Application monitoring samples will be collected by LRC staff and analyzed by Eurofins to verify the amount of dicamba applied. Four sets of four Whatman #3 filter paper (12.5 cm diameter) replicates (16 total samples) will be placed systematically throughout the application area and at canopy height. The location and height of each set of application monitoring samples will be documented in the field notebook.

As soon as possible after application, each filter paper in a set will be carefully folded and placed into a uniquely labeled container. Each container will be sealed with a cap. The in-swath samples will be stored (while in the field) in closed insulated containers with dry or artificial ice, as soon as possible after collection. These samples must be kept separate (i.e., separate coolers), from all other samples during storage and shipping to avoid the potential for cross contamination. In-swath samples will then be stored and shipped in coolers containing dry ice or within approximately -20 °C mobile freezers until transferred to storage at the analytical laboratory at approximately -20°C prior to analysis.

#### **6.7.4 Spray Drift**

Spray drift deposition transects will be established perpendicular to the application area in all four directions (9 total transects; see Figure 1) with the filter papers placed at the following approximate distances from the edge of the application area: 3, 5, 10, 20, 40, 50, 60 m. An additional filter paper sample is to be placed up to approximately 90m from the application area for the three downwind



transects only, if there is enough space in the field to allow it. The field line will be defined as the outer edge of the spray from the furthest nozzle on the boom.

Sample collectors will be placed horizontally at canopy height with Whatman #1, 15 cm diameter filter papers that will be collected and placed in uniquely labeled containers following application of the homogenized tank mix and thereafter at set intervals.

Filter papers will be collected from each transect and distance at a nominal target time of approximately 1, 24, 48, 72, 96, 120, 144, and 168 hours (8 periods) following initiation of the application to the application area.

Deposition sample collection will be conducted by LRC staff and analyzed by Monsanto staff to measure the amount of dicamba deposited. Filter papers will be collected by starting with the furthest distance samples and working toward the application area to reduce the potential for cross contamination. The exact sampling scheme will be determined by the Field PI and documented in the field notebook.

#### **6.7.5 Field Volatility**

Sample collection will be conducted by LRC staff and analyzed by Eurofins to measure the amount of dicamba volatilized. Samples will be collected and placed in uniquely labeled containers. The sampling pumps beginning and end flow rates and times will be recorded for each sampling period and supplied to Modeling PI. In addition, any special circumstances during sample collection, such as PUF contamination, pump malfunction, broken tubes, etc., will be recorded and supplied to the Modeling PI.

#### **Pre-application Samples**

Two pre-application air samples (at 0.15 m above canopy height) will be collected using air sampling equipment placed near the in-field air monitoring location (center of the application area).

The pre-application samples will be collected 6 – 48 hours prior to the start of the application and will last for approximately 6 hours. These samples will be used to determine the level of background dicamba within the application area.

Pre-application samples will then be stored and shipped in coolers containing dry ice or within approximately -20 °C mobile freezers until transferred to storage at the analytical laboratory at approximately -20°C prior to analysis.

#### **Post-application Samples**

Flux monitoring (field volatility) will be conducted with a single in-field air profile monitoring station (center mast) with collectors at five heights (approximately 0.15, 0.33, 0.55, 0.9, and 1.5 m above canopy height, with a duplicate sample at the 1.5m height). The in-field air samplers will be placed in the approximate center of the treated area (see Figure 1) and air sampling equipment turned on as soon as possible, but no later than 45 minutes, after completion of the application.

PUF samples at the center mast shall be collected from top to bottom to minimize contamination risk.

Eight monitoring stations will be located around the perimeter of the sprayed application area. Each perimeter station will be placed approximately 5 m outside of the application area and will have an air monitoring collector at 1.5 m above canopy height (Figure 1). The off-field air samplers will be covered during application to avoid contamination; covers will be removed, and air sampling equipment turned on as soon as possible, but no later than 45 minutes, after completion of the application. The open end of the perimeter PUF samplers will be positioned to face outward from the application area.

After application, both in-field and perimeter PUF samples will be collected at a nominal target time of approximately 6, 12, 24, 36, 48, 60, 72, 84, 96, 108, 120, 132, 144, 156, and 168 hours (15 periods) following completion of the application. Actual target sample collection times will be described by the Study Director at a later date, depending on sunrise/sunset times during the field phase of the study. At the center mast sampling station, all 6 samples from the previous period shall be collected before any of the PUFs for the following sampling period are attached to the pump, and the next sampling period is initiated.

After collection, post-application samples will be stored and shipped in coolers containing dry ice or within approximately -20 °C mobile freezers until transferred to storage at the analytical laboratory at approximately -20°C prior to analysis.

#### **6.7.6 Plant Effects**

Plant effects observations and measurements will be collected by Monsanto Company staff and statistically analyzed by Monsanto Company. Data will be collected in a field notebook. See Table 4 for an example data collection form.

Visual symptomology/plant response will be assessed on a scale of 0 to 100, with 0 representing no visible plant response and 100 representing complete plant death. This plant response rating scale will be conducted consistent with visual plant response ratings described in Frans and Talbert (1977). If different personnel are to evaluate plant symptomology, consistency in the visual ratings between individuals will be ensured by evaluating several plant response levels and cross-checking to verify that the independently estimated ratings are scored the same. For selected transects, distances, and timings, photographs will be made to document the visual plant response symptoms and severity at specified distances.

Plant height measurements will be taken by holding a plant upright and measuring the distance between the ground and the tip of the most recently emerged apical bud. Where multiple shoots are present, measurements along the main shoot will be taken. Measurements will be made to the nearest centimeter using a standard ruler. Plants will be selected non-systematically with no attempt to measure the same plant at subsequent time points.

A designated control plot will be established in the upwind portion of the field based on prevailing wind direction. Four locations representative of no observable visual dicamba symptomology within the control plot will be identified prior to application. These locations will be marked to enable returning to the same area for each time period.

Plant effects transects will be established perpendicular to the application area on all sides and diagonally at the corners of the application area to assess both primary and secondary off-site movement (spray drift) of approximately 60 m (197 feet), and up to approximately 90m (295 feet) in the downwind direction if there is enough space in the field to allow it (13 total transects; see Figure 1). To assess secondary movement only (volatility), transects will be established on all sides of the application area using tarps (volatility covers) of approximately 20 m (66 feet) before application, during application, and following application for at least 30 minutes to prevent exposure to spray drift (9 total transects; see Figure 1). The field line (start of the off-field transects) will be defined as the outer edge of the spray from the furthest nozzle on the boom. Plant effects transects (spray drift and volatility) will be paired with a spray drift deposition transect and the transects will be established along the edge of the treated areas as to adequately cover the length of the field edge. Untarped plant effects transects will extend out to 90m in the downwind direction and in the two adjacent diagonal directions. The remaining 6 transects will extend to 60m (see Figure 1). Transects will not be located within pre-determined designated ingress and egress areas for the sprayer. Any additional observations outside of the established transects will be noted in the field notebook; imagery will be retained in the study file.

### Controls

Visual symptomology rating and plant heights will be measured at approximately 0, 14, and 28 Days After Treatment (DAT) from four designated locations representative of no observable visual dicamba symptomology prior to application. Each location will have one aggregate visual symptomology score with ten plant height measurements (4 visual symptomology [VS] and 40 plant height [PH] total observations per time point).

### 0 DAT

One visual symptomology rating and ten plant heights will be measured within each transect (22 total transects) from edge of field at 3 locations for spray drift transects (0-90 m range) and 2 locations for volatility transects (0-20 m range) to ensure of no prior plant injury and to provide a measure of inherent variability in the plant sizes prior to application (57 VS and 570 PH total observations).

### 14 DAT and 28 DAT

One visual symptomology rating and ten plant heights will be measured within each transect (22 total transects) from each designated distance from edge of field (3, 5, 10, 20, 40, 50, 60, 90 m,  $\pm 1$  row/meter for spray drift transects and 3, 5, 10, 20 m  $\pm 1$  row/meter for volatility transects) (132 VS and 1320 PH total observations per time point).

### 6.7.7 Quality Monitoring Samples

Quality monitoring samples, Field Exposed Spikes and Transit Stability samples, will be shipped from Eurofins (PUFs) and Monsanto Company (filter papers) overnight on dry ice under chain of custody to:

Chris Schaubert  
27928 Sandoval Road  
Shattuc, IL 62231

### Field Exposed Spikes

Field exposed spikes will be spiked in the lab at Eurofins and shipped on dry ice to the field site. Field exposed spikes (spiked PUFs) will be weathered for approximately 6 and 12 hours to determine the amount, if any, of dicamba lost during a sampling event. Weathering will consist of drawing air across the field exposed spikes in the same manner as the flux monitoring samples except that they will be placed where no additional exposure to test material is expected to occur. For both time intervals there will be 3 replicates of PUFs fortified at 4 concentrations: 0, 3, 10, and 30 ng dicamba/PUF for a total of 24 field exposed spike samples. Each individual sample will have a unique identifier. After collection, the samples will be stored and shipped on dry ice separately from study samples to avoid contamination.

### Transit Stability Samples

Transit stability samples will be prepared in the Monsanto laboratory (filter paper) and Eurofins laboratory (PUF) and shipped on dry ice to and from the field site to determine stability of dicamba during transport. The samples will be stored on dry ice or in approximately -20C freezers upon receipt at the field site. Chain of custody documents will be completed, and the transit stability samples will be shipped from Shattuc, IL to Eurofins (PUFs) and Monsanto (filter papers) by means representative of how the field samples will be delivered to the laboratories (e.g. shipment on dry ice or mobile -20C Freezers).

Transit stability samples will consist of 3 replicates of PUFs fortified at 2 concentrations: 0 and 30 ng dicamba/PUF for a total of 6 PUF transit stability samples and 5 replicates of filter paper (FP) fortified at 2 concentrations: 0 and 0.05 µg dicamba/filter paper for a total of 10 FP transit stability samples.

## 6.8 Sample Handling, Storage, and Disposal

PUF and filter paper samples will be handled with gloves (to be replaced after the collection of samples from each sampling station) and placed in conical tubes labeled with unique sample identification information. These labels will contain unique sample identification codes.

Field volatility, spray drift, application monitoring, field exposed spikes, and transit samples will be kept in frozen storage (e.g., coolers containing dry ice or -20C freezes) prior to and during shipment. Upon receipt at the analytical laboratories, samples will be kept in frozen storage at approximately -20°C prior to analysis.

Tank mix samples will be stored and shipped under ambient conditions.

All samples will be shipped by overnight courier or delivered by LRC staff under chain of custody.

Ship all high-concentration samples (application monitoring filter paper and tank mix) and PUF samples (pre-application, post-application, field exposed spikes and transit stability) to:

Matt Rebstock  
Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202  
Phone: +1-573-777-6385  
E-mail: [MatthewRebstock@eurofinsUS.com](mailto:MatthewRebstock@eurofinsUS.com)

Ship all trace filter paper samples (spray deposition and transit stability) to:

Michael R. Shepard, Ph.D.  
c/o Chris Weber  
Monsanto Company  
700 Chesterfield Parkway West  
GGA & GGB Dock (FF2939)  
Chesterfield MO 63017  
Phone: +1-636-737-9332  
E-mail: [michael.r.shepard.jr@monsanto.com](mailto:michael.r.shepard.jr@monsanto.com)

Sample storage conditions, chain-of-custody records, and disposal methods will be documented in the study file.

## **6.9 Weather Data Collection**

### **Spray Drift**

Before the experiment is conducted, a Site Meteorological Station will be erected on site. Wind speed and direction at the time of application will be recorded using the same sonic anemometer associated with the Site Meteorological Station described below.

An anemometer will be placed at field edge, downwind from the application area at boom height to collect additional wind speed and direction data during application.

### **Field Volatility**

#### **Site Meteorological Station and Data Collection**

During field volatility sample collection, the following environmental conditions will be recorded at the main meteorological station within proximity of the test plot (see Table below). The location will be approved by the Study Director.

The following parameters will be recorded at the intervals listed (Collection Periods), or more frequently, and reported in the Field Sub-Report at the intervals specified (Reporting Periods). The heights listed in the below table are nominal values, and the actual heights are to be documented in the field notebook and field sub-report.

#### Site Meteorological Data Descriptions

Parameter	Monitoring Height/Depth (from ground surface)	Collection Periods (minimum interval)	Reporting Periods
Air Temperature	1.7 m, 5 m, 10 m	Minute	Hourly
Wind Speed and Direction*	1.7 m, 5 m, 10 m	Minute	Hourly
Relative Humidity	1.7 m	Minute	Hourly
Precipitation	1.7 m	Minute	Daily
Solar Radiation	1.7 m	Minute	Hourly
Soil Temperature	1 mm, 2-inch, 6-inch depths	Minute	Hourly
Soil Moisture	2-inch depth	Minute	Hourly

\*If data is collected at 1-minute intervals, statistics summarizing the 1-second intervals (minimum, maximum, average, and standard deviation) will also be collected in the raw data.

#### Flux Monitoring Meteorological Station and Data Collection

A primary flux meteorological station will be established approximately 100ft from the center of the application area after application so that a good representation of the wind pattern near the center mast is achieved (Figure 1). The station will be positioned crosswind to the center mast so that it is at an approximate right angle to the prevailing wind direction from the center mast. The station will monitor air temperature, wind speed and wind direction (using sonic anemometers) located approximately 0.33, 0.55, 0.90 and 1.5 m above canopy height.

There will be an identical, secondary flux meteorological station established near the field edge as a contingency. The flux met station near the center of the application area will be the primary source of met data for modelling purposes while the field edge met station will be a secondary source of met data, used only in the event it is needed (e.g., data gap from primary source as it is translocated near center of application area). Source of met data will be identified in a final report.

The following parameters will be recorded at the intervals listed (Collection Periods), or more frequently, and reported in the Field Sub-Report at the intervals specified (Reporting Periods).

**Flux Monitoring Meteorological Data Descriptions**

Parameter	Monitoring Height (m above the crop canopy)	Collection Periods (minimum interval)	Reporting Periods
Air temperature	0.33, 0.55, 0.90, 1.5	Minute	Hourly
Wind speed and Direction	0.33, 0.55, 0.90, 1.5	Minute	Hourly

**6.10 Return of Unused Test Substance and Tank Mix Partners**

Any unused test substance and/or tank mix partner that is marked experimental use only will be packaged appropriately and returned to:

Carolina Santangelo  
Sample Processing Coordinator - Q2C/Q212A  
Monsanto Company  
800 N. Lindbergh Blvd.  
St. Louis, MO 63167  
Phone: 314-694-5088  
Email: carolina.santangelo@bayer.com

The shipment container must be clearly marked with the following information:

**Sponsor Study Number: REG-2019-0035**

**7.0 Analytical Methods****7.1 Determination of Dicamba from Pre-application, Post-application, Field Exposed, and Transit Monitoring Samples**

Dicamba collected via polyurethane foam (PUF) will be analyzed according to analytical method ME-1902 or ME-2242 using the version(s) current at the time of initiation of sample analysis. Any modifications to the method(s) will be documented in the study file. Acceptance criteria defined in the current version(s) of the respective analytical methods ME-1902 or ME-2242 will be followed.

Dicamba collected via Whatman #1 filter paper will be analyzed according to analytical method ME-1871 using the version current at the time of initiation of sample analysis. Any modifications to the method will be documented in the study file. Acceptance criteria defined in the current version of analytical method ME-1871 will be followed.

## 7.2 Determination of Dicamba from Application Monitoring Samples

Sample preparation and the method for analysis of collected in-swath samples following deposition of dicamba will follow the current version of analytical method ME-2166 for the determination of dicamba after deposition on filter paper. Any modifications to the method will be documented in the study file. Acceptance criteria defined in the current version of ME-2166 will be followed.

Tank mix sample preparation and the method(s) for analysis of collected samples will follow the current version of the analytical method ME-2154, with modification to measure pH, for the determination of dicamba in each treatment. Any additional modifications to the method will be documented in the study file. Acceptance criteria defined in the current version of analytical method ME-2154 will be followed.

## 7.3 Analytical Sub-reports

Analytical chemistry sub-reports, which includes a QA statement and GLP Compliance Statement, will be prepared by each Analytical Chemistry Principal Investigator. The final sub-reports will be prepared in accordance with PR Notice 2011-3, and will contain all the information required by, and will be handled per, 40 CFR § 160.185, as applicable.

Analytical results for Pre-application, Application, Post-application, In-swath, Tank Mix, and quality monitoring samples will be provided in their respective sub-reports prepared by their respective Analytical Chemistry Principal Investigators. The Laboratory Quality Control samples with recoveries outside the range listed by the respective method will be discussed in the sub-reports.

## 8.0 Control of Bias

### Contamination

Bias and cross-contamination will be controlled by thoroughly cleaning spray equipment prior to application and with detailed procedures for obtaining representative samples while avoiding contamination.

Filter papers will be collected by starting with the furthest distance samples and working toward the application area. Nitrile, or other protective gloves, will be changed before collecting samples at a given distance.

The center mast sample team will use appropriate PPE for entering the application area and will change protective gloves before collecting the PUF sample. Sample collection will start with the highest collector and work to the lowest.

Perimeter PUF samplers will be positioned to face away from the application area to reduce the risk of contamination due to movement of dicamba off field for reasons other than volatility.



Field volatility tarps will be removed carefully in such a way as to minimize contamination and damage to the tarped soybeans. Tarps will be removed starting with the edge furthest from the application area and working toward the application area. The covered transects are not to be located immediately adjacent to the deposition transects to avoid any potential influence on downwind air movement.

High level samples (application monitoring and tank mix) and trace level samples (all others) will be kept separate while stored in the field and when shipped and upon receipt at the laboratories.

### **Observations**

If multiple personnel are to evaluate plant symptomology, consistency in the visual ratings between individuals will be ensured by evaluating several different plant response levels and cross-checking to verify that the independently estimated ratings are scored the same. Personnel that collect plant ratings will confer in the field before final ratings are scored.

## **9.0 Statistical Methods**

### **Spray Drift**

Statistical analyses of the spray drift deposition samples will be conducted by Monsanto Company staff and results reported.

Non-linear regression will be used to model deposition (mean or 90<sup>th</sup> percentile values) for all time periods and in all four directions. Any other statistical methods used for analysis of the data will be described in a final report.

An estimated 'no-effect distance' will be determined in all four directions based on the distance of the regression to reach the no observed effect rate (NOER; i.e., 0.000522 fraction of applied at target application rate of 0.5 lb dicamba a.e./A) for the most sensitive species from the guideline vegetative vigor study (Porch 2009).

### **Volatile Flux and Off-target Deposition and Air Concentration Modeling**

Statistical analyses of the field volatility samples and meteorological data will be conducted by Monsanto staff using the Aerodynamic flux (AD) and Integrated Horizontal Flux (IHF) methods and by Exponent staff using the Indirect (ID) flux method. Analyses will include the regression of air concentration, air temperature, and wind speed (dependent variables) as a function of the natural logarithm of height (independent variable).

All air samples will be used for the regression analysis unless samples are disqualified by breakage or other criteria. These criteria may include: 1) data obtained during special aspects/events of the trial history (such as severe weather events) that exhibit anomalous values, or 2) data that fails a statistically valid outlier test, or 3) data points that in the professional judgement of the researchers can be justifiably discarded for other possible reasons. In addition, dicamba air concentrations resulting from analytical results below the LOD (defined as 30% of the LOQ) will not be used for any downstream analysis, however those between LOD and LOQ will be evaluated for suitability

by inspecting the log-linear fit against sample height. Any other statistical methods used for analysis of the data will be described in the modeling sub-report.

The conceptual basis of the flux models assumes certain conditions in the field (including crop height or closure), which may necessitate that the IHF method be excluded. Justification for any data or flux model omission will be clearly articulated to the Study Director for final approval. For each sampling time increment, a mass rate of chemical transfer per unit surface area per unit time (i.e. flux) will be calculated. A minimum of 3 points are required for statistical analysis.

Each flux calculation method stated above will result in a time-varying flux profile. Exponent staff will develop a conservative flux profile using data from the three flux profiles (AD, IHF, and ID) along with meteorological data from three representative locations to model off-target deposition and air concentrations using appropriate air dispersion models (e.g. AERMOD, PERFUM). These deposition and air concentrations will provide a conservative estimate of potential off-target movement of volatile material under a variety of environmental conditions. Further, these deposition and air concentration estimates will be compared to a NOER and NOAEC, respectively.

### **Plant Effects**

Summary statistics (means and standard deviations) will be calculated for visual symptomology and plant height at each distance in all directions, including diagonals, both for tarped and untarped transects. The raw data may be presented graphically to aid in the characterization of off-site movement.

Non-linear regression will be used to estimate a 'no-effect distance' for each transect (plant height values). A linear mixed model will be used to compare plant heights in tarped versus untarped transects in each direction at each distance 28 DAT. Pairwise comparisons will be made at the  $\alpha = 0.05$  level. Any other statistical methods used for analysis of the data will be described in a final report.

### **10.0 Records to be Maintained**

All field records related to this study, including all raw data, the protocol, deviations, amendments, relevant correspondence, sub-reports and final report will be retained in the respective Study Director's or Principal Investigators' archives. Analytical raw data will be archived at the completion of the study by the analytical laboratories, Eurofins and Monsanto. Each Principal Investigator will provide the Study Director with documentation of the dates of archiving.

Archive-to-archive transfers of all data (original or verified copy) to the Monsanto Regulatory Archives will be coordinated by the respective Principal Investigator within a reasonable timeframe after completion of the study. Each Principal Investigator will provide the Study Director with documentation of the date of the archive-to-archive transfer to the Monsanto Regulatory Archives.

Archival transfers will be addressed to:

Suzanne Shoemaker FF2428J  
Monsanto Company  
2067 Westport Center Drive  
St Louis, MO 63146

## **11.0 Changes to the Protocol**

### **11.1 Protocol Amendments**

Any planned change to this protocol must be approved by the Study Director and the Sponsor Representative prior to making the change and will be documented as a protocol amendment.

### **11.2 Protocol and SOP Deviations**

Any unplanned change to this protocol and applicable SOPs will be documented as a deviation. If necessary, the Study Director will discuss the issue with the Sponsor Representative. The Study Director will determine the appropriate action and acknowledge the deviation. Actions taken and acknowledgments will be documented with a dated signature.

## **12.0 Final Report**

A final report, which includes a QA statement and GLP Compliance Statement, will be prepared by the Study Director, or designee. The final report will be prepared in accordance with PR Notice 2011-3, and will contain all the information required by, and will be handled per, 40 CFR § 160.185, including a description of the activities of this study and an assessment of the quality of all data and procedures required by this protocol.

## **13.0 References**

- American Society of Agricultural and Biological Engineers (ASABE) Standard. Revised 2013. Procedure for Measuring Drift Deposits from Ground, Orchards, and Aerial Sprayers. ASAE S561.1 APR2004 (R2013).
- Frans, R.E. and Talbert, R.E. 1977. Design of field experiments and the measurement and analysis of plant responses. In B. Truelove (Ed.), Research Methods in Weed Science (Second Edition), Southern Weed Science Society, Auburn University, Alabama (1977), pp. 15-23.
- International Standard (ISO). 2005. Equipment for crop protection – Methods for field measurement of spray drift. ISO 22866. 1<sup>st</sup> ed.
- Porch J.R., Krueger H.O., Kendall T.Z., Holmes C. 2009. BAS 183 09 H (Clarity): A Toxicity Test to Determine the Effects of the Test Substance on Vegetative Vigor of Ten Species of Plants. BASF Study No: 358586. MRID 47815102.

- U.S. Environmental Protection Agency. 1998. Office of Prevention, Pesticides and Toxic Substances (OPPTS); Spray Drift Test Guidelines, OPPTS 840.1200 Spray Drift Field Deposition. March. EPA 712-C-98-112.
- U.S. Environmental Protection Agency. 2008. Office of Prevention, Pesticides and Toxic Substances (OPPTS); Fate, Transport and Transformation Test Guidelines. OPPTS 835.8100 Field Volatility. October. EPA 712-C-08-024.
- U.S. Environmental Protection Agency. Revised 2016. U.S. EPA Generic Verification Protocol for Testing Pesticide Application Spray Drift Reduction Technologies for Row and Field Crops. June.
- U.S. Environmental Protection Agency. 2018. Office of Pesticide Programs, Registration Division communication to Thomas Marvin, Bayer CropScience, regarding Notice of Pesticide Registration of M1768 Herbicide, dated November 1, 2018.

## 14.0 Tables

**Table 1. Treatment List**

Treatment Number	Test Substance + Tank Mix Partners (rates)	Nozzle
1	MON 76980 (22 oz/A) + MON 79789 (32 oz/A) + Intact™ (0.5% v/v) + MON 51817 (1% v/v)	TTI 11004

**Table 2. Samples to be Collected**

Field Volatility Study Samples				
Sample Type [Matrix]	# of Samples	# of Time Points	# of Replicates	Study Total
Pre-application [PUF]	1	1	2	2
Off-field Perimeter [PUF]	8	15	1	120
In-field Center mast [PUF]	5, 1 duplicate	15	1	90
In-Swath [FP]*	4	1	4	16
Tank mix [Liquid]**	3	2	1	6
Field Exposed [PUF]	4	2	3	24
Transit Stability [PUF]	2	1	3	6
*In-Swath: 1 per quadrant (4 locations) x 4 FP per location			TOTAL	264
**Tank Mix: 3 pre-application + 3 post-application				

Field Deposition Study Samples				
Sample Type [Matrix]	# of Distances	# of Time Points	# of Replicates	Study Total
Upwind [FP]	7	8	2	112
Downwind [FP]	8	8	3	192
Left [FP]	7	8	2	112
Right [FP]	7	8	2	112
Transit Stability [FP]	2	1	5	10
			TOTAL	538

Soil and Water Characterization				
Sample Type [Matrix]	# of Samples	# of Time Points*	# of Replicates	Study Total
Source water	1	1	1	1
Soil	1 (15 cores)	1	1	1
*To be collected before application			TOTAL	2

**Table 2. Continued**

Plant Effects Observations and Measurements							
	# of Distances or Locations	# of Visual Symptomology/Distance or Location	# of Plant Heights/Distance or Location	# of Transects (Untarped)	# of Transects (Tarped)	Total Visual Symptomology Observations	Total Plant Height Observations
Control Plot – 0, 14, 28 DAT							
Control	4	1	10	n/a	n/a	4	40
Volatility Transects – 0 DAT							
Upwind	2*	1	10	n/a	2	4	40
Downwind	2*	1	10	n/a	3	6	60
Left	2*	1	10	n/a	2	4	40
Right	2*	1	10	n/a	2	4	40
Volatility Transects – 14 DAT and 28 DAT							
Upwind	4	1	10	n/a	2	8	80
Downwind	4	1	10	n/a	3	12	120
Left	4	1	10	n/a	2	8	80
Right	4	1	10	n/a	2	8	80
Spray Drift Transects – 0 DAT							
Upwind	3*	1	10	2	n/a	6	60
Downwind	3*	1	10	3	n/a	9	90
Left	3*	1	10	2	n/a	6	60
Right	3*	1	10	2	n/a	6	60
Diagonal	3*	1	10	4	n/a	12	120
Spray Drift – 14 DAT and 28 DAT							
Upwind	7	1	10	2	n/a	14	140
Downwind	8	1	10	3	n/a	24	240
Left	7	1	10	2	n/a	14	140
Right	7	1	10	2	n/a	14	140
Diagonal	7 (8 upwind)	1	10	4	n/a	30	300
*Non-systematic locations within each designated transect [0-90 m for spray drift transects and 0-20 m for volatility transects]							

**Table 3. Sampling Locations and Schedule**

<b>Polyurethane Foam [PUF]</b>			
<b>Sample Type, Locations</b>	<b>Height (m above crop canopy)</b>	<b>Sample Intervals</b>	<b>Total</b>
<b>Pre-application, 2 replicates</b>	0.15	6 – 48 hr before application	2
<b>Center Mast, 1 location</b>	0.15, 0.33, 0.55, 0.9, 1.5	0-6	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	6-12	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	12-24	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	24-36	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	36-48	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	48-60	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	60-72	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	72-84	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	84-96	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	96-108	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	108-120	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	120-132	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	132-144	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	144-156	5, 1 dup
	0.15, 0.33, 0.55, 0.9, 1.5	156-168	5, 1 dup
<b>Off-field Perimeter, 8 locations</b>	1.5	0-6	8
	1.5	6-12	8
	1.5	12-24	8
	1.5	24-36	8
	1.5	36-48	8
	1.5	48-60	8
	1.5	60-72	8
	1.5	72-84	8
	1.5	84-96	8
	1.5	96-108	8
	1.5	108-120	8
	1.5	120-132	8
	1.5	132-144	8
	1.5	144-156	8
	1.5	156-168	8

*Table 3. Continued*

<b>Filter Paper [FP]</b>			
<b>Locations</b>	<b>Distances from edge of field (m)</b>	<b>Sample Intervals</b>	<b>Total</b>
<b>Upwind, 2 transects</b>	3, 5, 10, 20, 40, 50, 60	0-1	7
	3, 5, 10, 20, 40, 50, 60	1-24	7
	3, 5, 10, 20, 40, 50, 60	24-48	7
	3, 5, 10, 20, 40, 50, 60	48-72	7
	3, 5, 10, 20, 40, 50, 60	72-96	7
	3, 5, 10, 20, 40, 50, 60	96-120	7
	3, 5, 10, 20, 40, 50, 60	120-144	7
	3, 5, 10, 20, 40, 50, 60	144-168	7
<b>Downwind, 3 transects</b>	3, 5, 10, 20, 40, 50, 60, 90	0-1	8
	3, 5, 10, 20, 40, 50, 60, 90	1-24	8
	3, 5, 10, 20, 40, 50, 60, 90	24-48	8
	3, 5, 10, 20, 40, 50, 60, 90	48-72	8
	3, 5, 10, 20, 40, 50, 60, 90	72-96	8
	3, 5, 10, 20, 40, 50, 60, 90	96-120	8
	3, 5, 10, 20, 40, 50, 60, 90	120-144	8
	3, 5, 10, 20, 40, 50, 60, 90	144-168	8
<b>Left, 2 transects</b>	3, 5, 10, 20, 40, 50, 60	0-1	7
	3, 5, 10, 20, 40, 50, 60	1-24	7
	3, 5, 10, 20, 40, 50, 60	24-48	7
	3, 5, 10, 20, 40, 50, 60	48-72	7
	3, 5, 10, 20, 40, 50, 60	72-96	7
	3, 5, 10, 20, 40, 50, 60	96-120	7
	3, 5, 10, 20, 40, 50, 60	120-144	7
	3, 5, 10, 20, 40, 50, 60	144-168	7
<b>Right, 2 transects</b>	3, 5, 10, 20, 40, 50, 60	0-1	7
	3, 5, 10, 20, 40, 50, 60	1-24	7
	3, 5, 10, 20, 40, 50, 60	24-48	7
	3, 5, 10, 20, 40, 50, 60	48-72	7
	3, 5, 10, 20, 40, 50, 60	72-96	7
	3, 5, 10, 20, 40, 50, 60	96-120	7
	3, 5, 10, 20, 40, 50, 60	120-144	7
	3, 5, 10, 20, 40, 50, 60	144-168	7



**Table 4. Example Plant Height and Visual Injury Rating Data Sheet (0 DAT, 14 DAT, or 28 DAT)**

Date	DAT	Transect ID	Distance (m)	Visual Injury (%)	Plant Height (cm)									
					1	2	3	4	5	6	7	8	9	10
		Volatility UWA	3											
			5											
			10											
			20											
		Drift UWA	3											
			5											
			10											
			20											
			40											
			50											
			60											

DAT – days after treatment

Signature

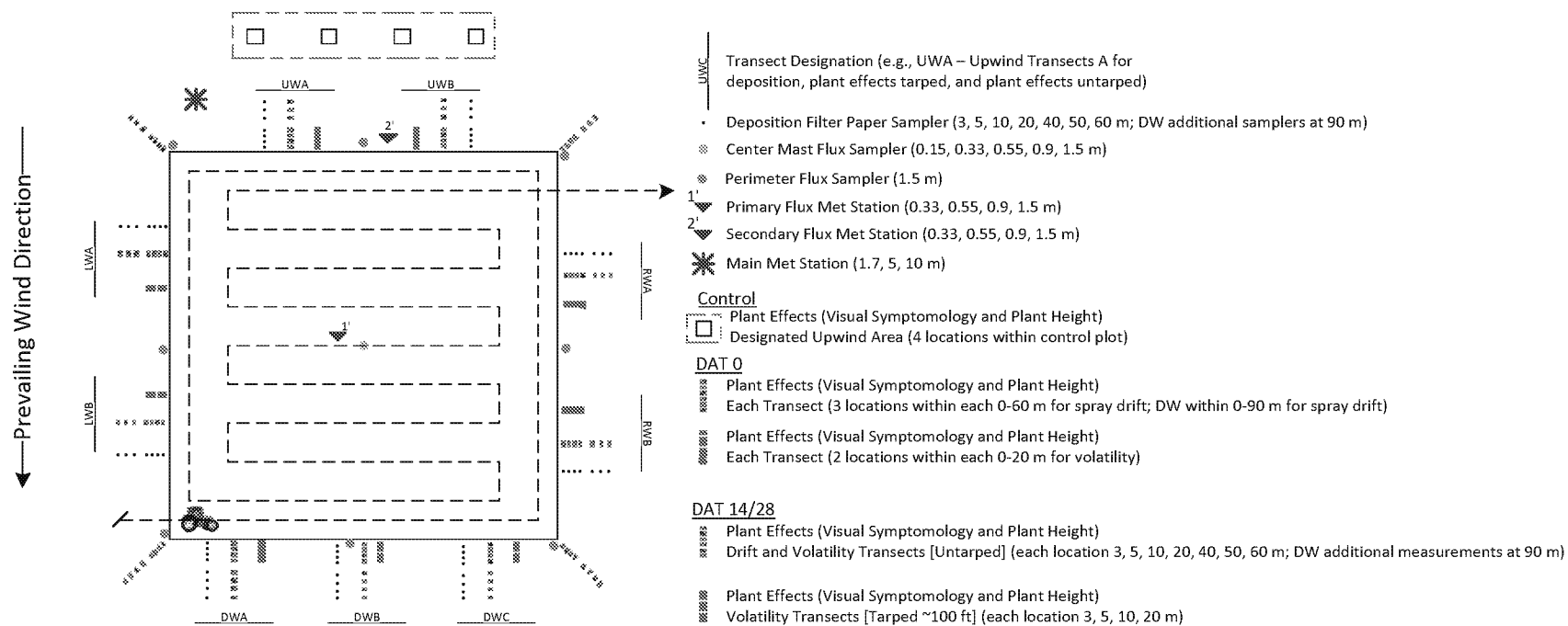
Date

Signature

Date

## 15.0 Figures

**Figure 1. Representative Site Layout**



## PROTOCOL CHANGE DOCUMENT NO. 1

**Study Title** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

**Sponsor Study No.** REG-2019-0035

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 6/18/2019

**Description of Change:** The protocol is being amended so that the first paragraph of section 6.7.6 Plant Effects reads as follows.

Plant effects observations and measurements will be collected by Monsanto Company staff and statistically analyzed by Monsanto Company. Data will be collected in the field notebook at approximately 0, 14, and 28 days after treatment (DAT; +/- 2 days; actual dates will be recorded in the field notebook). See Table 4 for an example data collection form.

**Reason for Change:** This text was modified to allow for a +/- 2 day window from the nominal collection time for plant effects data.

**Effect of Change on Study:** No adverse effect on study.

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 6/18/2019

**Description of Change:** The protocol is being amended so that the second paragraph of section 6.9 Weather Data Collection reads as follows.

### **Spray Drift**

An anemometer will be placed at approximately 3 m off-field edge, downwind from the application area at boom height to collect additional wind speed and direction data during application.

## PROTOCOL CHANGE DOCUMENT NO. 1

**Reason for Change:** This text was modified to add specificity as to the approximate placement of the boom-height anemometer (i.e. 3 m off-field edge.)

**Effect of Change on Study:** No adverse effect on study.

---

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 6/18/2019

**Description of Change:** The protocol is being amended so that the following information is added to the end of section 6.9 Weather Data Collection.

### **Plant Effects Meteorological Station and Data Collection**

During the plant effects data collection phase of the study (approximately -1DAT to 28DAT), the following environmental conditions will be recorded at the plant effects meteorological station within proximity of the test plot (see Table below). The location will be approved by the Study Director.

The following parameters will be recorded at the intervals listed (Collection Periods), or more frequently, and presented in the Plant Effects Field Sub-Report at the intervals specified (Reporting Periods). The heights listed in the below table are nominal values, and the actual heights are to be documented in the field notebook and field sub-report.

## PROTOCOL CHANGE DOCUMENT NO. 1

### Plant Effects Meteorological Data Descriptions

Parameter	Monitoring Height/Depth (from ground surface)	Collection Periods (minimum interval)	Reporting Periods
Air Temperature	1.7 m	Minute	Hourly
Wind Speed and Direction	1.7 m	Minute	Hourly
Relative Humidity	1.7 m	Minute	Hourly
Precipitation	1.7 m	Minute	Hourly
Solar Radiation	1.7 m	Minute	Hourly
Soil Temperature	1 mm	Minute	Hourly
Soil Moisture	0-6 in. depth	Minute	Hourly

**Reason for Change:** This information was included to ensure that proper metrological data is collected and reported for the entire duration of the Plant Effects Field Phase of the study.

**Effect of Change on Study:** No adverse effect on study.

---

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 6/18/2019

**Description of Change:** The protocol is being amended so that the first paragraph of section 9.0 Statistical Methods reads as follows.

#### Spray Drift

Statistical analyses of the spray drift deposition samples will be conducted by Monsanto Company staff and results reported. Dicamba concentrations resulting from analytical results below the LOD (defined as 30% of the LOQ) will not be used for any downstream analysis, however those between LOD and LOQ will be evaluated for suitability by the Statistical Analyses Principal Investigator for Deposition.

## PROTOCOL CHANGE DOCUMENT NO. 1

**Reason for Change:** This text was added to provided clarity regarding the use of sub-LOQ samples in the Statistics and Modeling Phase of the study.

**Effect of Change on Study:** No adverse effect on study.

---

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 6/18/2019

**Description of Change:** The protocol is being amended to include Kendall Zuber as the recipient of the trace filter paper samples found in section 6.8 Sample Handling, Storage, and Disposal. The protocol now reads as follows.

Ship all trace filter paper samples (spray deposition and transit stability) to:

Michael R. Shepard, Ph.D.  
c/o Kendall Zuber  
Monsanto Company  
700 Chesterfield Parkway West  
GGA & GGB Dock (FF2939)  
Chesterfield MO 63017  
Phone: +1-636-737-9332  
E-mail: [michael.r.shepard.jr@monsanto.com](mailto:michael.r.shepard.jr@monsanto.com)

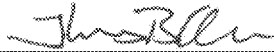
**Reason for Change:** Personnel change occurred at Monsanto Company.

**Effect of Change on Study:** No adverse effect on study.

---

PROTOCOL CHANGE DOCUMENT NO. 1

Approved By:



Thomas B. Orr

Sponsor Representative

Monsanto Company

6/18/2019

Date



Will J. Griesse

Study Director

Monsanto Company

6/18/2019

Date

## PROTOCOL CHANGE DOCUMENT NO. 2

**Study Title** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

**Study No.** REG-2019-0035

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 8/13/2019

**Description of Change:** The protocol is being amended so that the “Spray Drift” subsection of section “9.0 Statistical Methods” reads as follows:

### **Spray Drift**

Statistical analyses of the spray drift deposition samples will be conducted by Monsanto Company staff and results reported.

Non-linear regression will be used to model deposition for all time periods and independently for each filter paper transect in all four directions. Any other statistical methods used for analysis of the data will be described in a final report.

An estimated ‘no-effect distance’ will be determined independently for each filter paper transect in all four directions based on the distance of the regression to reach the no observed effect rate (NOER; i.e., 0.000522 fraction of applied at target application rate of 0.5 lb dicamba a.e./A) for the most sensitive species from the guideline vegetative vigor study (Porch 2009).

**Reason for Change:** This text was modified to indicate that each transect of spray drift deposition samples will be modeled independently rather than as part of a group consisting of all filter paper transects found on each of the four sides of the application area.

**Effect of Change on Study:** No adverse effect on study.

---



## PROTOCOL CHANGE DOCUMENT NO. 2

---

☒ Amendment

☐ Deviation

**Effective Date:** 6/28/2019

**Description of Change:** The protocol is being amended so that the shipping address specified in section “6.7.7 Quality Monitoring Samples” reads as follows.

Chris Schaubert  
27928 County Rd 1300 N  
Shattuc, IL 62231

**Reason for Change:** This text was modified to provide a more accurate shipping address for the Quality Monitoring Samples, as the initially provided address could be confused with another location.

**Effect of Change on Study:** No adverse effect on study.

---

☒ Amendment

☐ Deviation

**Effective Date:** 7/1/2019

**Description of Change:** Section 6.7 of the protocol is being amended to allow for the use of coolers containing artificial ice, instead of dry ice, for the transport of samples from the field to the -20°C storage freezers aboard the Lange Research mobile lab.

**Reason for Change:** The protocol previously only specifically mentioned the use of artificial ice for In-Swath Samples. This change was made to allow for its use with all sample types found in this study.

**Effect of Change on Study:** No adverse effect on study.

---

## PROTOCOL CHANGE DOCUMENT NO. 2

---

☒ Amendment

☐ Deviation

**Effective Date:** 7/1/2019

**Description of Change:** The protocol is being amended so that the “Preparation of Tank Mix” sub-section of section “6.6 Test Substance Application” includes the following statement.

If the tank mix is prepared but application is delayed until the following day, the tank mix does not need to be disposed of and remixed if it is kept under constant agitation overnight.

**Reason for Change:** This text was added to allow for the preservation of the tank mix under certain conditions, should a delay to application occur.

**Effect of Change on Study:** No adverse effect on study.

---

☒ Amendment

☐ Deviation

**Effective Date:** 7/1/2019

**Description of Change:** The protocol is being amended so that an additional sampling location will be added to the Left-Wind B transects at 90m for both the drift deposition filter paper transect and the drift plant effects transect.

**Reason for Change:** In anticipation of wind blowing from the southwest direction during application, these distances were added to transects located near the northeast side of the application area in order to collect data out to 90m in the downwind direction.

**Effect of Change on Study:** No adverse effect on study.

---

## PROTOCOL CHANGE DOCUMENT NO. 2

---

☒ Amendment

☐ Deviation

Effective Date: 7/1/2019

**Description of Change:** The protocol is being amended so that the second paragraph of section "6.9 Weather Data Collection" reads as follows.

An anemometer will be placed at approximately 3 m off-field edge, upwind from the application area at boom height to collect additional wind speed and direction data during application.

**Reason for Change:** The text was modified to allow for the placement of the boom height anemometer on the upwind side of the application area, as opposed to the downwind side.

**Effect of Change on Study:** No adverse effect on study.

---

**Approved By:**



Thomas B. Orr  
Sponsor Representative  
Monsanto Company

8/13/19

Date



Will J. Gries  
Study Director  
Monsanto Company

8/13/19

Date

### PROTOCOL CHANGE DOCUMENT NO. 3

**Study Title** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

**Sponsor** REG-2019-0035  
**Study No.**

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 10/7/2019

**Description of Change:** The protocol is being amended so that the second paragraph of the “Plant Effects” sub-section of section “9.0 Statistical Methods” reads as follows.

Non-linear regression will be used to estimate a ‘no-effect distance’ **for each transect (plant height values); this formal analysis of plant height values will only be conducted in transects where visual symptomology exceeds 10% (at any distance).** A linear mixed model will be used to compare plant heights in tarped versus untarped transects in each direction at each distance 28 DAT. Pairwise comparisons will be made at the  $\alpha = 0.05$  level. Any other statistical methods used for analysis of the data will be described in a final report.

**Reason for Change:** By definition, when visual symptomology  $\leq 10\%$ , the level of dicamba exposure is not sufficient to impact plant growth and therefore the ‘no-effect distance’ based on plant height is 0 m.

**Effect of Change on Study:** No adverse effect on study.

---

PROTOCOL CHANGE DOCUMENT NO. 3

Approved By:

Sponsor approval documented via associated email. WJG  
Thomas B. Orr 10-7-19  
Date

Sponsor Representative  
Monsanto Company



Will J. Griesse  
Study Director  
Monsanto Company

10/7/2019  
Date

**PROTOCOL DEVIATION**

**Study Title:** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 - Illinois

**Monsanto Study No.:** REG-2019-0035

**Document Number:** 4

**Effective Date:** Date of Study Director's Signature

---

**1. Description of Deviation:** Protocol section – “6.7.1 Soil and Source Water Characterization”

The soil characterization sample was shipped chilled instead of ambient.

**Reason for Deviation:** Researcher oversight.

**Effect of Deviation on Study:** This deviation is not expected to have a negative effect on the study.

**2. Description of Deviation:** Protocol section – “6.7.1 Soil and Source Water Characterization”

Water holding capacity for 15 bar (disturbed) was not requested or performed for soil characterization.

**Reason for Deviation:** Researcher oversight.

**Effect of Change on Study:** This deviation is not expected to have a negative effect on the study.

**3. Description of Deviation:** Protocol section – “6.7.1 Soil and Source Water Characterization”

The following parameters were not measured in a Series 3 water characterization but would have been measured in a Series 5 test: potassium, carbonate, bicarbonate, nitrate, sulfate, chloride, and alkalinity. The following parameters were not measured in a Series 5 but were measured in a Series 3 test: total dissolved solids, and turbidity.

**Reason for Deviation:** Researcher oversight.

**Effect of Change on Study:** This deviation is not expected to have a negative effect on the study.

**4. Description of Deviation:** Protocol section – “6.6 Test Substance Application”

The tractor sprayer speed was not calibrated 3 times prior to application.

**Reason for Deviation:** Researcher oversight.

**Effect of Change on Study:** This deviation is not expected to have a negative effect on the study as the sprayer was within an average of 3% of target pass times.

**5. Description of Deviation:** Protocol section – “6.7.4 Spray Drift”

The following samples were unable to be collected.

Sample No	Study	Type	Location	Distance	Timing
1006901	LRC-2019-0035	FPR	DWA	10M	96HR
1006903	LRC-2019-0035	FPR	DWA	40M	96HR
1006908	LRC-2019-0035	FPR	DWA	5M	120HR
1006910	LRC-2019-0035	FPR	DWA	20M	120HR
1006977	LRC-2019-0035	FPR	DWB	60M	120HR
1006990	LRC-2019-0035	FPR	DWB	20M	168HR
1007040	LRC-2019-0035	FPR	DWC	50M	120HR
1007041	LRC-2019-0035	FPR	DWC	60M	120HR
1007087	LRC-2019-0035	FPR	LWA	3M	96HR
1007089	LRC-2019-0035	FPR	LWA	10M	96HR
1007090	LRC-2019-0035	FPR	LWA	20M	96HR
1007097	LRC-2019-0035	FPR	LWA	20M	120HR
1007145	LRC-2019-0035	FPR	LWB	10M	96HR
1007146	LRC-2019-0035	FPR	LWB	20M	96HR
1007147	LRC-2019-0035	FPR	LWB	40M	96HR
1007148	LRC-2019-0035	FPR	LWB	50M	96HR
1007149	LRC-2019-0035	FPR	LWB	60M	96HR
1007150	LRC-2019-0035	FPR	LWB	3M	120HR
1007152	LRC-2019-0035	FPR	LWB	10M	120HR
1007154	LRC-2019-0035	FPR	LWB	40M	120HR
1007156	LRC-2019-0035	FPR	LWB	60M	120HR
1007167	LRC-2019-0035	FPR	LWB	20M	168HR
1007200	LRC-2019-0035	FPR	RWA	5M	96HR
1007202	LRC-2019-0035	FPR	RWA	20M	96HR
1007203	LRC-2019-0035	FPR	RWA	40M	96HR
1007204	LRC-2019-0035	FPR	RWA	50M	96HR
1007205	LRC-2019-0035	FPR	RWA	60M	96HR

1007210	LRC-2019-0035	FPR	RWA	40M	120HR
1007226	LRC-2019-0035	FPR	RWA	60M	168HR
1007256	LRC-2019-0035	FPR	RWB	5M	96HR
1007261	LRC-2019-0035	FPR	RWB	60M	96HR
1007262	LRC-2019-0035	FPR	RWB	3M	120HR
1007263	LRC-2019-0035	FPR	RWB	5M	120HR
1007266	LRC-2019-0035	FPR	RWB	40M	120HR
1007267	LRC-2019-0035	FPR	RWB	50M	120HR
1007268	LRC-2019-0035	FPR	RWB	60M	120HR
1007324	LRC-2019-0035	FPR	UWA	20M	96HR
1007325	LRC-2019-0035	FPR	UWA	40M	96HR
1007326	LRC-2019-0035	FPR	UWA	50M	96HR
1007327	LRC-2019-0035	FPR	UWA	60M	96HR
1007328	LRC-2019-0035	FPR	UWA	3M	120HR
1007329	LRC-2019-0035	FPR	UWA	5M	120HR
1007331	LRC-2019-0035	FPR	UWA	20M	120HR
1007334	LRC-2019-0035	FPR	UWA	60M	120HR
1007377	LRC-2019-0035	FPR	UWB	3M	96HR
1007380	LRC-2019-0035	FPR	UWB	20M	96HR
1007384	LRC-2019-0035	FPR	UWB	3M	120HR

**Reason for Deviation:** Due to rain some deposition filter papers were unable to be collected.

**Effect of Deviation on Study:** This deviation expected to have minimum negative effect on the study.

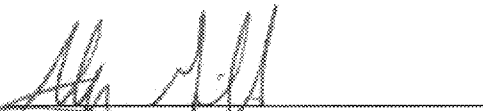
Approved by:

Study Director, Monsanto Company

  
Will J. Giese

2/25/2020  
Date

Field Research Principal Investigator, Lange Research and Consulting, Inc.

  
Alex Gibbs  
Scientist

02/20/2020  
Date



**PROTOCOL DEVIATION**

**Study Title:** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + IntactTM + MON 51817 - Illinois

**Monsanto Study No.:** REG-2019-0035

**Document Number:** 5

**Effective Date:** March 18, 2020

**1. Description of Deviation:** Protocol section – “6.5 Test Substance Storage and Container Handling”

The temperature of the test substance was not recorded during temporary storage from June 19, 2019 to June 30, 2019, prior to test substance application.

**Reason for Deviation:** Researcher oversight.

**Effect of Deviation on Study:** Due to tank mix verification samples and application verification filter paper samples showing dicamba concentrations within the expected range, this deviation is not expected to have a negative effect on the study.

Approved by

Study Director, Monsanto Company

  
Will J. Griesse

03/18/2020  
Date

## PROTOCOL CHANGE DOCUMENT NO. 6

**Study Title** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 – Illinois

**Sponsor** REG-2019-0035  
**Study No.**

---

☒ **Amendment**

☐ **Deviation**

**Effective Date:** 3/27/2020

**Description of Change:**

Pertaining to protocol Section “10.0 Records to be Maintained”

Exponent will transfer all relevant study data and reports to the Study Director, which will then be archived directly into the Monsanto Company archives.

**Reason for Change:** Exponent Inc. is a non-GLP facility and so does not have archives managed under GLPs. Therefore, an archive-to-archive transfer from Exponent to Monsanto cannot be achieved.

**Effect of Change on Study:** No adverse effect on study.

---

PROTOCOL CHANGE DOCUMENT NO. 6

Approved By:

Approved Via Email

Thomas B. Orr

Sponsor Representative

Monsanto Company

March 27, 2020

Date



Will J. Griesse

Study Director

Monsanto Company

3-27-2020

Date

## **Appendix B. Lange Research and Consulting, Inc. Field Sub-Report**

Off-target Movement Assessment of a Spray Solution Containing  
MON 76980 + MON 79789 + Intact™ + MON 51817 - Illinois

Volatility and Deposition Field Sub-Report

**TEST SUBSTANCE:** MON 76980

**DATA REQUIREMENT(S):** US EPA OPPTS 840.1200: Spray Drift Field  
Deposition  
US EPA OPPTS 835.8100: Field Volatility

**AUTHOR(S):** Alex Gibbs

**REPORT COMPLETION DATE:** March 13, 2020

**SPONSOR/PRIMARY TEST FACILITY:** Monsanto Company  
700 Chesterfield Parkway West  
Chesterfield, MO 63017

**TEST FACILITY:** Lange Research and Consulting, Inc  
4746 W. Jennifer Ave. Suite 105  
Fresno, CA 93722

AGVISE Laboratories  
604 Highway 15 West  
P.O. Box 510  
Northwood, ND 58267

**MONSANTO STUDY ID:** REG-2019-0035  
**LANGE STUDY ID:** LR19397  
**AGVISE STUDY ID:** 19-1371 (soil) and 19-115 (water)

**Total pages: 72**

**© 2020 Bayer Group. All Rights Reserved.**

*This document is protected under national and international copyright law and treaties. This document and any accompanying material are for use only by the regulatory authority to which it has been submitted by Monsanto Company and its affiliates, collectively "Bayer Group", and only in support of actions requested by Bayer Group. Any other use, copying, or transmission, including internet posting, of this document and the materials described in or accompanying this document, without prior consent of Monsanto Company, is strictly prohibited; except that Monsanto Company hereby grants such consent to the regulatory authority where required under applicable law or regulation. The intellectual property, information and materials described in or accompanying this document are owned by Bayer Group, which has filed for or been granted patents on those materials. By submitting this document and any accompanying materials, Monsanto Company and the Bayer Group do not grant any party or entity any right or license to the information, material or intellectual property described or contained in this submission.*

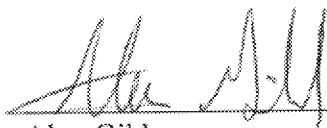
## GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

The field portion of this study was conducted in accordance with the United States EPA Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) Good Laboratory Practice (GLP) Standards (40 CFR 160), with the following exceptions:

Field Phase:


(The following information does not require generation under the GLPs (40 CFR Part 160) as long as it is clearly stated as such: weather data collection, soil characterization, general land preparation (i.e., tillage, mowing, irrigation, etc.), application of maintenance chemicals during the study, and documentation of pesticide use history.)

1. Test site observations such as estimation of slope.
2. Pesticide and crop histories for test plots.
3. Soil taxonomy information provided by the USDA Natural Resources Conservation Service (NRCS).
4. Test plot preparation prior to application.
5. SpotOn nozzle calibration devices used to calibrate the sprayer not maintained under GLP and no SOP in place. SpotOn calibrators were verified prior to use. Verification was documented in the raw data.
6. GPS Coordinates



Alex Gibbs

Field Research Principal Investigator  
Lange Research and Consulting, Inc.



Date

## QUALITY ASSURANCE STATEMENT

**Study Title:** Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 - Illinois

**Study No.:** REG-2019-0035

Audit inspection reports have been submitted to the Study Director, Testing Facility Management, and Principal Investigator documenting the status of compliance with applicable departmental standard operating procedures, the study protocol, and Good Laboratory Practice regulations.

Lange Research and Consulting (LRC) (QA) was responsible for providing QA oversight for this phase of the study. LRC designated QA performed audits and inspections for the field portion of this study and assured that the sub-report accurately describes the methods and SOPs, and that the reported results accurately reflect the raw data for the field portion of the study (site preparation, test substance application, a component of each measurement event over the course of the study, field notebook, and raw field data). Audits and inspections are listed below pursuant to Good Laboratory Practice Regulations (40 CFR Part 160).

AGVISE Laboratories QAU performed inspections/audits of the soil and water characterization procedures and raw data. Audits and inspections are listed below pursuant to Good Laboratory Practice Regulations (40 CFR Part 160).

Inspection Dates	Phase Audited	Date Reported to Study Director and Study Director Management
June 30, 2019	Field Audit – Plot Set Up	12 July 2019
June 30-July 1-2, 2019	Field Audit – Calibration, Tank Mix, and Application	12 July 2019
June 30- July 2, 2019	Field Audit – Sampling	03 July 2019
July 2, 2019	Field Audit – Sample Collection	12 July 2019
August 6-7, 2019	Data Audit – Sample Tables for Filter Papers and Field Samples	18 August 2019
August 7-9, 2019	Data Audit – Weather Data	18 August 2019
July 19, 2019	AGVISE Procedure Audit– pH of Water	22 August 2019
July 30, 2019	AGVISE Procedure Audit - pH Analytical Procedure	22 August 2019
August 7, 2019	AGVISE – Raw Data Audit	22 August 2019



**QUALITY ASSURANCE STATEMENT (continued)**

Inspection Dates	Phase Audited	Date Reported to Study Director and Study Director Management
November 6-7, 10, 2019	Data Audit – Field Trial Notebook	03 December 2019
December 21, 23-24, 2019	Draft Report Audit	08 January 2020
February 20, 2020	Final Report Audit	20 February 2020

In compliance with the Good Laboratory Practice regulations, this report has been reviewed by Quality Assurance.

Quality Assurance Unit:

Carla M Knipp Date: 13 Mar 2020  
Carla Knipp  
Knipp Consulting LLC - Quality Assurance

## GENERAL INFORMATION

### Contributors

The following personnel contributed to the conduct of this study and the Field Sub-Report in the capacities indicated:

**Name:****Role:****Monsanto Company**

Thomas B. Orr, M.S.

Will Griesse

Rodrigo Sala, Ph.D.

Michael R. Shepard, Ph.D.

Sponsor Representative

Study Director

Primary Testing Facility Management

Testing Facility – Analytical Chemistry Principal Investigator

**AGVISE Laboratories**

Larry Wikoff

Testing Facility – Soil and Water Characterization  
Principal Investigator**Test Site**

Chris Schaubert

Schaubert Farms

Field Cooperator

**Lange Research and Consulting**

Alex Gibbs

Field Research Principal Investigator – Volatility and Deposition

**Study Dates**

Study Initiation Date:

31-May-2019

Experimental Start Date:  
(application date)

02-Jul-2019

Experimental Termination Date:  
(Deposition/Volatility Sampling  
Phase Completion Date)

09-Jul-2019

**Retention of Raw Data**

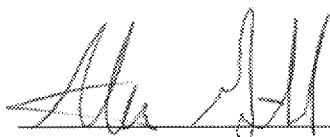
The original field phase report and study-specific raw data generated by LRC will be temporarily archived at LRC upon completion of the study phase. The original field phase report and raw data will be subsequently transferred to the Monsanto Regulatory Archives, St. Louis, Missouri, USA for final archiving. Copies of the field phase report and the study-specific raw data will be archived at LRC together with all appropriate non-study specific supporting data.

## REPORT APPROVAL

**Report Title:**

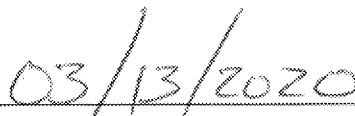
Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact<sup>TM</sup> + MON 51817 - Illinois

This sub-report is an accurate and complete representation of the field portion of study activities.

**Field Research Principal Investigator:**

Alex Gibbs

Lange Research and Consulting, Inc.



Date

## ABBREVIATIONS AND SYMBOLS

<b>Abbreviation</b>	<b>Definition</b>
A	Acre
a.e.	acid equivalent
a.i.	active ingredient
°C	Degrees Celsius
cm	centimeter(s)
COA	Certificate of Analysis
DAT	Days After Treatment
°F	Degrees Fahrenheit
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
ft	foot, feet
g	gram(s)
gal	gallon(s)
GLP	Good Laboratory Practice
GPA	Gallons Per Acre
GPM	Gallons Per Minute
GPS	Global Positioning System
ha	hectare(s)
hh:mm	hour: minute
hr, hrs	hour(s)
in	inch(es)
km	kilometer(s)
L	Liter(s)
lb	pound(s)
LRC	Lange Research and Consulting, Inc.
m	meter(s)
µg	microgram(s)
mEq	milliequivalent(s)
mL	milliliter(s)
mm	millimeter(s)
mmhos	Millimhos
min	minute(s)
Monsanto	Monsanto Company
mph	miles per hour
ng	nanogram(s)
nm	nanometer(s)
ND	Non-detect
NRCS	USDA Natural Resources Conservation Service

OTT	Over-The-Top (application to a cropped plot)
oz	ounce(s)
ppm	parts per million
psi	pounds per square inch
PUF	polyurethane foam
QA	Quality Assurance
s, sec	Second
std dev	standard deviation
SOP	Standard Operating Procedure
TTI	Turbo TeeJet Induction (® TeeJet Technologies, Spraying Systems Co.)
USDA	United States Department of Agriculture
US EPA	United States Environmental Protection Agency
µg	Microgram
UV	Ultraviolet
W	Watts
w/w	weight per weight

## TABLE OF CONTENTS

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT.....	3
QUALITY ASSURANCE STATEMENT .....	4
GENERAL INFORMATION.....	6
REPORT APPROVAL .....	7
ABBREVIATIONS AND SYMBOLS.....	8
TABLE OF CONTENTS.....	10
<b>1.0</b> STUDY OBJECTIVE.....	13
<b>2.0</b> METHODS AND MATERIALS.....	13
<b>2.1</b> Site Selection.....	13
<b>2.2</b> Experimental Design and Test System.....	13
<b>2.3</b> Study Management.....	14
<b>2.4</b> Test and Reference Substances .....	14
<b>2.4.1</b> Test Substances.....	14
<b>2.4.2</b> Tank Mix Partners.....	15
<b>2.4.3</b> Reference Substance .....	16
<b>2.5</b> Test Site and Test Plot.....	16
<b>2.5.1</b> Site Selection .....	16
<b>2.5.2</b> Site Location .....	17
<b>2.5.3</b> Test Plot Dimensions and Design .....	17
<b>2.5.4</b> Test Plot Layout and Instrumentation.....	18
<b>2.6</b> Test Substance Application Equipment and Verification .....	18
<b>2.7</b> Test Plot Crop and Pesticide History .....	19
<b>2.8</b> Tank Mix Preparation and Spray Application.....	19
<b>2.9</b> Weather Data Collection .....	20
<b>2.9.1</b> 10-Meter Main Meteorological Station .....	20
<b>2.9.2</b> Boom Height Anemometer .....	20
<b>2.9.3</b> Long Duration Main Meteorological Station.....	21
<b>2.9.4</b> Primary Flux Meteorological Station.....	21
<b>2.9.5</b> Secondary Flux Meteorological Station.....	21
<b>2.10</b> Test Plot Soil and Water Characterization .....	22

2.11	Sample Handling, Storage, and Disposition .....	22
2.12	Tank Mix Samples.....	23
2.13	Application Verification Spray Area Samples .....	23
2.14	Flux Monitoring Samples (Field Volatility).....	24
2.14.1	Pre-Application Samples .....	24
2.14.2	Post-Application Samples.....	24
2.15	Spray Drift Samples.....	25
2.16	Quality Control Samples .....	26
2.16.1	Field Exposed Spikes (PUF Collectors) .....	26
2.16.2	Transit Stability (PUF Collectors and Filter Paper).....	26
2.17	Crop Destruct.....	27
2.18	Control of Bias.....	27
2.19	Deviations from Protocol.....	27
3.0	RESULTS .....	28
3.1	Test Substance Spray Rate Verification and Spray Application.....	28
3.2	Test Site Environmental Monitoring (During and After Application).....	28
3.3	Test Plot Soil and Water Characterization .....	29
3.4	Tank Mix Samples .....	29
3.5	Application Verification Filter Paper Samples .....	29
3.6	Flux Monitoring Samples (Field Volatility).....	29
3.6.1	Pre-Application Samples .....	29
3.6.2	Post-Application Samples.....	29
3.6.3	Transit Stability Air Samples.....	30
3.6.4	Field Exposed Spikes.....	30
3.7	Spray Drift Deposition Samples.....	30
3.7.1	Transit Stability Filter Paper Samples .....	30
3.8	Plant Effects Samples.....	30
4.0	CONCLUSIONS.....	30
5.0	REFERENCES .....	32
6.0	TABLES .....	33
7.0	FIGURES.....	40
8.0	APPENDICES .....	52

## List of Tables

Table 1. AGVISE Soil Characteristics.....	34
Table 2. AGVISE Water Characteristics .....	35
Table 3. Meteorological Conditions During Spray Application.....	36
Table 4. Period Statistics for Wind Data from Main Meteorological Station .....	37
Table 5. Period Statistics for Temperature and Relative Humidity Data from Main Meteorological Station.....	38
Table 6. Spray Application Verification Information.....	39

## List of Figures

Figure 1. Study Site.....	41
Figure 2. Test Plot Diagram.....	42
Figure 3. Test Site Location Relative to the Field Cooperator's Shop in Shattuc, IL .....	43
Figure 4. Ground Boom Sprayer.....	44
Figure 5. 10-Meter Main Meteorological Station .....	45
Figure 6. Boom Height Anemometer.....	46
Figure 7. Flux Meteorological Station.....	47
Figure 8. Spray Drift Deposition Sampling Platform .....	48
Figure 9. In-Field PUF Sampling Station .....	49
Figure 10. Perimeter PUF Sampling Station.....	50
Figure 11. Plant Effects-Volatilization Sampling Area .....	51

## List of Appendices

Appendix A. Certificate of Analysis MON 76980 and MON 79789 .....	53
Appendix B. Agronomic and Pesticide Use History .....	56
Appendix C. USDA NRCS Clinton County, IL Soil Report.....	57
Appendix D. AGVISE Soil and Water Characterization Reports .....	61
Appendix E. Sample Period Collection Dates and Times .....	64



## 1.0 STUDY OBJECTIVE

The objective of this study was to determine off-target movement due to volatility and spray drift and resulting impacts to non-target plants (via volatility and drift) of a Monsanto dicamba herbicide formulation.

Data from this study provide support for the registration and stewardship of typical end-use pesticide products. The purpose of this sub-report is to describe the field phase set up, sampling, and meteorological data collection for further downstream assessments.

## 2.0 METHODS AND MATERIALS

The materials and methods used in this study are described below and draw from methods described in US EPA (2016), ISO (2005), ASABE (revised 2013), US EPA Spray Drift Test Guideline, OPPTS 840.1200 Spray Drift Field Deposition (1998), and US EPA Fate, Transport, and Transformation Test Guideline, OPPTS 835.8100 Field Volatility (2008). Additional detailed method information is provided in separate sub-reports as appendices to the main report, along with the study protocol and protocol changes.

### 2.1 Site Selection

The field location was selected to represent a typical commercial scale application to a post-emergent soybean field. The study was conducted near Shattuc, Illinois. The application area was approximately 293 m by 296 m (960 ft by 970 ft ) (approximately 21 acres) located within an approximate 117-acre field. The 21-acre application area was planted with dicamba-tolerant soybean while the surrounding area was planted with non-dicamba tolerant soybean. Both tolerant and non-tolerant soybeans were planted on June 3, 2019 in 0.51 meter (20 inch) rows. The test plot was oriented within the larger 117-acre field to minimize any potential wind obstructions (buildings, hedgerow, etc.) and was located at least approximately 305 m (1,000 ft) away from other anticipated dicamba applications within  $\pm 1$  week of the application.

### 2.2 Experimental Design and Test System

The study was performed in Clinton County, near Shattuc, IL (38.667391°, -89.15170303°). The test system included one agricultural field planted with both dicamba tolerant and non-tolerant soybean (Figure 1). The designated test plot was an exposed area with no obstructions that could influence air flow in the areas of application or measurement. The test plot was identified with at least one plot corner identifier labeled with the protocol number.

## 2.3 Study Management

Monsanto Company (Chesterfield, Missouri) was the study sponsor and provided study directorship and primary test facility management for this study. Lange Research and Consulting (LRC, Fresno, CA) managed the volatility and deposition field phase of this study in partnership with Monsanto Company. Monsanto Company managed the plant effects field phase of this study. Chris Schaubert (Shattuc, IL) was the field co-operator, provided the land and spray equipment for the study, managed the spray applications for the study and performed the application under the supervision of LRC. LRC and Monsanto provided GLP-trained field staff for equipment calibration, site instrumentation, and collection of samples during the field phase of the study. Eurofins, Monsanto, and AGVISE Laboratories performed the analytical phases of the study. Exponent Inc. and Monsanto conducted the statistical analysis and modeling phase of the study.

## 2.4 Test and Reference Substances

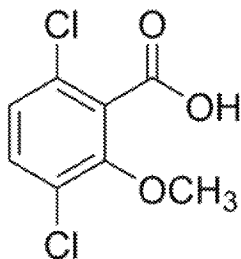
### 2.4.1 Test Substances

The test substance used in the field phase of this study was MON 76980. The formulation MON 76980 contains dicamba in the form of its diglycolamine salt (nominally 42.8% by weight, 29.0% a.e.). In addition to the test substance, three tank mix partners were used: MON 79789 (glyphosate potassium salt), Intact™ (polyethylene glycol, choline chloride, guar gum), and MON 51817 (potassium acetate).

The active ingredient dicamba is an herbicide registered in the United States for selective control of broadleaf weeds in several crop and non-crop use patterns. The formulation tested was developed for post-emergence (over-the-top) control of broad-leaf weeds (including hard-to-control weeds) in soybean with the dicamba tolerance trait.

Dicamba Molecular Formula:  $C_8H_6Cl_2O_3$

Dicamba Chemical Structure:



### MON 76980

EPA Registration Number:	524-617
Active Ingredients:	Dicamba (diglycolamine salt), 29.0% (a.e.)
Actual a.e. Content (COA):	28.7% wt. (a.e.)

CAS Number: 104040-79-1  
Lot Number: 11495284  
Formulation Type: Liquid  
Expiration Date (COA): May 10, 2020  
Density (COA): 1.1971 g/mL  
pH: ~5.3

The concentrated test substance was characterized by the Sponsor. A copy of the Certificate of Analysis (COA) for the test substance was sent to LRC and retained in the records sent to the Study Director (Appendix A).

The test substance was provided by Monsanto and shipped to the LRC facility in Moberly, MO, from where it was then transported to the test site. The test substance was received from the Sponsor on May 14, 2019. Upon receipt, the test substance was logged in and stored in a chemical storage room and then transported to test site in Shattuc, IL at ambient temperatures ranging from approximately 51.8 to 97.5 °F prior to application on July 2, 2019. All records pertaining to receipt, storage and transport temperatures were monitored and maximum and minimum temperatures were recorded by LRC personnel. Temperatures were not recorded from June 19 to 30, 2019.

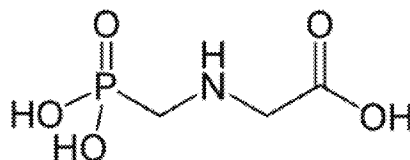
#### 2.4.2 Tank Mix Partners

##### MON 79789

EPA Registration Number: 524-549  
Active Ingredients: Glyphosate (monoethanolamine salt), 39.8% (a.e.)  
Actual a.e. Content (COA): 39.7% wt. (a.e.)  
CAS Number: 70901-12-1  
Lot Number: 11495283  
Formulation Type: Liquid  
Expiration Date (COA): May 7, 2020  
Density (COA): 1.3526 g/mL  
pH: 4.3-4.8

Glyphosate Molecular Formula:  $C_3H_8NO_5P$

Glyphosate Chemical Structure:



##### Intact™

WA Registration Number: 9349-16001

Principal Functioning Agents:	Polyethylene glycol, choline chloride, guar gum
Labeled PFA Content:	43.18%
CAS Number:	25322-68-3 (Polyethylene glycol)
Formulation Type:	Liquid
Expiration Date:	14-May-2020
Density:	8.93 lbs/gallon
pH:	6.5 to 8.5

**MON 51817**

EPA Registration Number:	NA
Active Ingredients:	Potassium acetate
Nominal Content:	50% wt.
CAS Number:	127-08-2
Lot Number:	390116
Formulation Type:	Liquid
Expiration Date:	14-May-2020
Density:	1.2 g/cm <sup>3</sup>
pH:	6

The tank mix partners used were provided by Monsanto and shipped to the LRC facility in Moberly, MO, from where they were then transported to the test site. They were received from the Sponsor on May 14, 2019. Upon receipt, the tank mix partners were logged in and stored in a chemical storage room and then transported to test site in Shattuc, IL at ambient temperatures ranging from approximately 51.8 to 97.5 °F prior to application on July 2, 2019. All records pertaining to receipt, storage and transport temperatures were monitored and maximum and minimum temperatures were recorded by LRC personnel. Temperatures were not recorded from June 19 to 30, 2019.

**2.4.3 Reference Substance**

No reference substance was applied in the field. Dicamba analytical reference standards used in the analytical phase of the study are listed in the Analytical Sub-Report (an Appendix to the Main Report).

**2.5 Test Site and Test Plot****2.5.1 Site Selection**

The site selected by the Study Director in coordination with the Field Principal Investigator met the following criteria:

- Field with sufficient size for a soybean test plot with approximate minimum 300-ft no-spray buffer zones around the plot and at least 1,000 feet away from other anticipated dicamba applications within the same week as sample collection
- Minimal wind obstruction of the test plots
- Less than 1% average slope
- Sufficient expected temperatures and humidity for efficacious field volatility and deposition sampling

The agricultural field meeting the criteria listed above was identified through consultation with the field cooperator and was located near Shattuc, IL. The state of Illinois and Clinton County represent a key soybean growing and high dicamba use region. The selected farm field represented a typical commercial setting for the application of the formulation being evaluated in this study for off-target movements. A site diagram is provided in Figure 2.

### 2.5.2 Site Location

The test field for the study was located on a farm owned by Chris Schaubert in Shattuc, IL and served as a central base of operations for the field phase of the study. This farm facility was where tank mixing occurred prior to test substance application.

The test site was uniform with respect to soil texture and vegetation, and near uniform regarding slope (<1%). Agronomic and pesticide use history was documented by the grower for the field in which the test site was located (Appendix B).

The only treatment for the study, referenced as Treatment 1 (MON 76980+MON 79789+Intact<sup>TM</sup>+MON 51817) throughout the text, was tolerant soybean-cropped test plot. The center point of the test plot was located at approximately 38.667391°, -89.151703°.

The Treatment 1 test site (Figure 1 and Figure 3) had the following characteristics:

- Located in a large, rectangular agricultural field, approximately 4.4 miles north of the farm facility with no windbreaks on any side, and an estimated slope <1%
- Soybean crop approximately 15 cm (6 inches) above the surface of crop rows within the plot

### 2.5.3 Test Plot Dimensions and Design

The test plot dimensions were approximately 960 by 970 ft for a total treated area of approximately 21 acres. Including the 300-ft buffers and surrounding field, the area occupied by the tolerant and non-tolerant soybean test plot was approximately 117 acres. During the field phase of the study, the condition of the test plot was documented with digital cameras. Aerial and other images of the test plots were included in the study raw data, when available.

The test plot and surrounding buffer zone were planted in tolerant (Asgrow AG41X8) and non-tolerant (Dyna-Gro S40GL59) soybean on June 3, 2019. The soybean seeds were planted at a density of 165,000 seeds/A on 20-inch row spacing. All planting was coordinated and documented by Chris Schaubert of Schaubert Farms, Shattuc, IL.

#### **2.5.4 Test Plot Layout and Instrumentation**

Prior to planting, the treated area was established and marked in the test site field using the GPS and a laser range finder so that the field cooperators knew where to plant the dicamba tolerant soybeans and non-tolerant soybeans. Prior to application, each test plot point of interest (i.e. spray drift sampling stations, volatility sampling stations, plant effects sampling stations (drift and volatility), plot corner, flux meteorological stations, spray swath midpoint, and application verification sampling locations) was navigated by using measuring tapes and a laser range finder and marked with survey flags. The orientation of test plot corresponded to the forecasted wind directions for July 2, 2019 Clinton County, Shattuc, IL.

Instrumentation consisted of off-target deposition sample collectors, air sampling (flux monitoring) equipment, transect plots (plant effects), meteorological monitoring stations, and spray swath sample collectors for application verification (AV), tank mix/source water samples, and a composited soil sample.

#### **2.6 Test Substance Application Equipment and Verification**

A self-propelled John Deere R4038 sprayer equipped with a 120 ft boom, 1,000 gallon tank, and Turbo TeeJet® Induction (TTI) 11004 nozzles manufactured by TeeJet Technologies were used for the spray application (Figure 4). Boom height was set to 20 inches above the crop canopy. A total of 96 nozzles at 15 in spacing were installed on the boom.

Sprayer/nozzle verification was performed on June 30, 2019 to verify spray consistency at the required pressure and rate and to test the nozzles to ensure none were plugged or defective resulting in an inconsistent manufacturer specification spray. TTI 11004 nozzle uniformity was tested by spraying water at a pressure of 63 psi through the boom. Nozzle output was measured using SpotOn® Model SC-1 sprayer calibrator devices (Innoquest, Inc.), and each nozzle was tested three times to determine variability.

The verification of the sprayer and the TTI 11004 nozzles established the total boom output per minute of spray to be 186.71 LPM (49.32 GPM). Using the measured volume per minute output of the boom at 63 psi, and a target speed of 13.6 mph, the target spray rate of 15.0 GPA for Treatment 1 was achieved (Table 6).

Due to the spray pattern required for this study the sprayer is required to start some passes from a stationary position, thus a constant sprayer speed is not possible. Variable flow rate technology

was used to adjust the sprayer output based on the sprayer speed, thus ensuring the sprayer output of 15.0 GPA regardless of speed.

## 2.7 Test Plot Crop and Pesticide History

The agronomic and pesticide use histories for the test field were obtained from the grower and documented for the four years preceding the study (2016, 2017, 2018, and 2019) by LRC. Grower records are included in the study file and are also provided in Appendix B.

## 2.8 Tank Mix Preparation and Spray Application

Before the test substance tank mix was created, the sprayer tank and lines were emptied, then triple-rinsed with water. Tank mixes were then prepared at 9:12 am at Schaubert Farms facility located approximately 4.4 miles south of the test site.

The tank mix was prepared on July 1, 2019, by first adding 382.78 gal (1449.0 L) of water to the clean, empty sprayer tank. Intact (7,600 ml) was added next, followed by MON 51817 (15,150 ml), MON 76980 (17,350 ml), and MON 79789 (25,250 ml). The tank mix was completed at approximately 9:20 am. Prior to tank mix application, wind direction was observed by the Study Director and the Field Research Principal Investigator to ensure wind direction criteria was met. It was determined that the wind was insufficient for application. A pump was placed in the sprayer to keep the tank mix agitated until application could be made the following day.

On July 2, 2019, the Study Director and Field Research Principal Investigator checked wind direction to ensure criteria was met. The default wind direction criteria based on plot orientation for this study was 225°. The application was made when the average wind direction was as close as possible to that target orientation, given the actual wind conditions on the day of application. The spray application was made during wind conditions of approximately 3-10 mph.

The Treatment 1 spray application was completed by an independent licensed pesticide applicator, Chris Schaubert from Schaubert Farms, on July 2, 2019. The homogenized tank mix of MON 76980 + MON 79789 + Intact<sup>TM</sup> + MON 51817 was applied at a target rate of 0.50 lb dicamba a.e./A.

For the application, Alex Gibbs of LRC rode in the cab of the sprayer with the applicator and recorded the spray swath pass times. Applications began only when the rolling 2-minute average windspeed was within the range of 3-10 mph.

Spray applications to the dicamba-tolerant and non-tolerant soybean test plot was representative of typical post-emergence herbicide applications to soybean (2-leaf stage or greater at time of application). The boom height for the application was set at 20 in (51 cm) above the soybean crop. Boom height was measured prior to the application at multiple locations along the boom and was adjusted to the target height as needed. For Treatment 1, the spray solution was applied

to an area, 21 acres (960 ft x 970 ft plot area), planted in dicamba-tolerant soybean. A 300-ft no-spray buffer area around the test plot was left unsprayed and contained non-dicamba tolerant soybean.

The application to the test plot on July 2, 2019 began at 9:35 am, consisted of perimeter plot spray swaths followed by interior swath passes (Figure 2), and concluded at 9:49 am (Table 3). The spray application was made with a target rate of 15.0 GPA and a target sprayer speed of 13.6 mph. Application verification calculated from pass times are provided in Table 6.

## **2.9 Weather Data Collection**

There were five meteorological stations used to collect data during various portions of the study.

### **2.9.1 10-Meter Main Meteorological Station**

The 10-Meter Main Meteorological Station was located upwind of the spray application area and measured wind speed and direction, and Temperature and Relative Humidity at three nominal heights: 1.7, 5, and 10 m. Wind speed and direction were measured using a Campbell Scientific CSAT 3D anemometer (model: CSAT3B) and two Campbell Scientific 2D anemometers (model: WindSonic1). Temperature and relative humidity were all measured using Campbell Scientific Temperature and Relative Humidity Sensor (model: HMP60). Solar Radiation was measured using Campbell Scientific Digital Thermopile Pyranometer (model: CS301). Precipitation was measured using Texas Instruments Rain Gauge (model: TE525). Soil Temperature at 1mm and 6 inches depth was measured using two Campbell Scientific Temperature Probe (model: 109). Soil Moisture and Temperature at 2 inches was measured using a Campbell Scientific Soil Probe (model: CS655). All sensors used in the 10-Meter Main Meteorological Station were connected to a Campbell Scientific CR1000X Datalogger. The 10-meter main meteorological sensors and the CR1000X Datalogger were used with a Campbell Scientific 4G Cellular Modem to remotely monitor data. The station was located upwind of the sprayed area (38.665533, -89.15325897; Figure 5).

### **2.9.2 Boom Height Anemometer**

The Boom Height Anemometer collected wind speed and wind direction data during test substance application at one nominal height of 20 inches above the crop canopy. Wind speed and direction were measured using a Campbell Scientific 2D anemometer (model: WindSonic1) and data was logged using a Campbell Scientific CR1000X Datalogger. The Boom Height Anemometer and the CR1000X Datalogger were used with a Campbell Scientific 4G Cellular Modem to remotely monitor data in real time. The station was located approximately 3 m upwind of the sprayed area (38.666051, -89.15252798; Figure 6).



### **2.9.3 Long Duration Main Meteorological Station**

The Long Duration Main Meteorological Station was located outside of the application area and recorded data for 28 days post-test substance application. The station consisted of one Campbell Scientific ClimaVUE sensor which measured wind speed/direction, air temperature, relative humidity, solar radiation, precipitation, and barometric pressure, (model: ClimaVUE50) located at 1.0 m; one Campbell Scientific soil moisture/temperature sensors (model: CS655). All sensors used in the Long Duration Main Meteorological Station were connected to a Campbell Scientific CR300 Datalogger. The Long Duration Main Meteorological sensors and the CR300 Datalogger were used with a Campbell Scientific 4G Cellular Modem to remotely monitor data. The Long Duration Main Meteorological Station was located upwind of the sprayed area (38.66595302, -89.15278103). Summary Data Tables from this meteorological station are provided in the Plant Effects Field Sub Report (Appendix of the main study report).

### **2.9.4 Primary Flux Meteorological Station**

The Primary Flux Meteorological Station was erected and deployed outside of the plot prior to test substance application. Following application, the Primary Flux Meteorological Station was moved to the center of the plot and stayed there until the morning of July 9, 2019, after the final drift sample was collected. The station consisted of four Campbell Scientific temperature sensors (model: 109) and four Campbell Scientific 2D anemometers (model: WindSonic1). The sensors were placed at four nominal heights: 0.33, 0.55, 0.9, and 1.5 m above crop canopy). All sensors used in the Primary Flux Meteorological Station were connected to a Campbell Scientific CR1000X Datalogger. The Primary Flux Meteorological sensors and the CR1000X Datalogger were used with a Campbell Scientific 4G Cellular Modem to remotely monitor data. The Primary Flux Meteorological Station was located in the middle of the plot (38.667374, -89.151524; Figure 7).

### **2.9.5 Secondary Flux Meteorological Station**

The Secondary Flux Meteorological Station was located upwind, outside of the sprayed area and recorded temperature, wind speed, and wind direction data at four nominal heights: 0.33, 0.55, 0.9, and 1.5 m above crop canopy. The station consisted of four Campbell Scientific temperature sensors (model: 109) and four Campbell Scientific 2D anemometers (model: WindSonic1). All sensors used in the Secondary Flux Meteorological Station were connected to a Campbell Scientific CR1000X Datalogger. The Secondary Flux Meteorological sensors and the CR1000X Datalogger were used with a Campbell Scientific 4G Cellular Modem to remotely monitor data. The Secondary Flux Meteorological Station was located upwind of the sprayed area (38.666022, -89.15217804).

## 2.10 Test Plot Soil and Water Characterization

Soil samples were collected from the test plot for characterization on June 30, 2019, one day before the anticipated spray applications. Fifteen core samples were extracted to a depth of 6 inches from across the test plot using a hand auger that was marked at 6 inches on the outside to ensure the proper sampling depth.

The fifteen core samples were composited by physically mixing the soil. A sub-sample of this composited soil was then placed into a one-gallon Ziploc® bag (at least 500 g of soil) and a pre-printed label was placed on the bag containing the following information: study number, site location, event description, date, and depth increment. The soil characterization sample was shipped chilled on blue ice to AGVISE Laboratories in Northwood, North Dakota for analysis. Soil characterization results are presented in Table 1 and Appendix D.

An NRCS Custom Soil Resource Report for Clinton County, Shattuc, IL was obtained from the Web Soil Survey website (<http://websoilsurvey.sc.egov.usda.gov/App/HomePage.htm>) and is presented in Appendix C.

A 1-L water sample was collected in a plastic container from the tank mix source water for characterization on June 30, 2019. The water was collected from the water source prior to test system tank mixing. The field pH of the sample was 8.17, measured at the time of collection using an Apera PH60 pH tester. The container for the water sample was labeled with the study number, site location, event description, date, and time period collected. The water characterization sample was shipped on ice to AGVISE Laboratories in Northwood, North Dakota for analysis. Water characterization results are presented in Table 2 and Appendix D.

## 2.11 Sample Handling, Storage, and Disposition

Application verification, PUF collector, deposition filter paper, and quality control samples were always handled with clean nitrile gloves, which were replaced after the collection of samples, prior to installation of a new sample media for the next sampling interval. PUF collector and filter paper samples were placed in conical tubes that were pre-labeled with unique sample identification information. Collected samples were checked in by LRC staff and either transferred to a freezer/cooler or stored in an ambient container for storage prior to shipping to the analytical test site.

Pre-application, application verification (in-swath), post-application, field exposed spikes, and transit stability PUF samples were stored in freezers prior to shipment. The Pre-application and application verification samples were stored in separate freezers from the post application, field exposed spikes, and transit stability samples. On July 10, 2019, these samples were hand delivered in ice chests containing dry ice to Eurofins, Columbia, MO.

Following collection deposition samples were stored in a freezer prior to shipment in ice chests containing dry ice to Monsanto Company, Chesterfield, MO.

Tank mix samples were stored and shipped under ambient conditions.

All samples, except soil and water, were hand delivered. The soil and water samples were shipped to AGVISE Laboratories, Northwood, ND by FedEx. The soil and water samples were shipped cool on blue ice.

The original chain-of-custody form accompanied each shipment of samples to the analytical laboratory. Each chain of custody included sample IDs for the samples included in that shipment, sampling date, and shipping date.

## **2.12 Tank Mix Samples**

Pre- and post-application samples were collected from the tank mix. These samples were analyzed for pH of the tank mix and to verify the amount of dicamba present in the tank mix solution. Tank mix samples were collected at two time points: (1) after the tank mix of the test substance had been sufficiently mixed, prior to application (pre-application), and (2) immediately following application (post-application). On July 1, 2019, three replicate samples of at least 50 mL each were collected using a 500 mL Nalgene bottle and placed into 120 mL amber glass jars. However, due to insufficient wind conditions, the application was delayed. After notifying the Study Director, the three samples were disposed, and new pre-application samples were taken the following day (July 2, 2019) prior to application. The application solution was agitated for 10 minutes and the new pre-application tank mix samples were collected directly into pre-labeled 120 mL amber glass jars with PTFE lined lids. The lids were placed onto the jars and sealed with electrical tape. The jars were double bagged in plastic re-closable bags. This was repeated for both time points for a total of 6 tank mix samples. After collection, these samples were kept ambient in temperature monitored storage, isolated from other samples, and shipped separately to avoid the potential for cross contamination. Following tank mix sample collection, the tank was emptied away from the study site location, and triple rinsed with water.

## **2.13 Application Verification Spray Area Samples**

Application verification sample collectors (In-swath/AV) consisted of four sets of four filter papers (Whatman #3, 12.5 cm diameter) affixed to disposable pieces of cardboard attached to a plexiglass tray on a pole set at the crop canopy height (6 in, 15 cm above the soil). Prior to test substance application, the four circular filter papers were placed on each of their respective sample collector platforms. At the test plot, a total of 4 groups of sample collectors (16 total samples) were placed across the application area. Distances from the collectors to the swath centers varied to capture various portions of the spray boom and different spray nozzles.

Immediately after the spray application was completed, the pads were removed and placed individually into pre-labeled 50 mL conical tubes. Each tube was capped, sealed with electrical tape and placed into a conical tube storage box. The box was taped shut and placed into a re-closable plastic bag. Application pads were immediately placed in an ice chest with artificial ice. Samples were transferred to frozen storage within 2 hours and stored separate from all other samples prior to shipping to the analytical test site.

## **2.14 Flux Monitoring Samples (Field Volatility)**

The SKC air pumps at the in-field monitoring station actively sampled for up to 168 hours after test substance application and were powered using 12v batteries. The sampling tubing and PUF collectors were protected from precipitation by ¾" diameter PVC pipes and the air sampling pumps were covered with plastic bags to protect from precipitation. Each pump was calibrated at the start of each monitoring period using an SKC Check-Mate 375-07550 calibrator. Pumps were calibrated to a target rate of 3.0 L/min and the flow rate was checked again at the end of each monitoring period.

### **2.14.1 Pre-Application Samples**

Two pre-application PUF collector air samples (from 0.15 m above the crop canopy) were collected on June 30, 2019 from 11:14 to 17:17, approximately 6 hours (Appendix E). Pre-application PUF collectors used air sampling equipment located at the approximate center of the test plot.

The pre-application samples were used to determine the potential levels of background dicamba within the application area. Following collection, pre-application samples were stored in freezers on site and shipped in an ice chest containing dry ice to the analytical test site.

### **2.14.2 Post-Application Samples**

The in-field air sampler used for monitoring flux for 168 hours following application, was placed in the approximate center of the test plot directly following spray application. The air sampling pumps at approximate heights of 0.15, 0.33, 0.55, 0.90, and two at 1.5 m above the crop surface were attached to a single center mast for the test plot and were turned on as soon as possible after completion of the application to the entire test plot (Figure 9).

After application, PUF collector samples were collected from the five established sampling heights on the sample mast at approximate nominal intervals of 6, 12, 24, 36, 48, 60, 72, 84, 96, 108, 120, 132, 144, 156, and 168 hours post-application. Sample collections for the 0-6 hour and 6-12 hour intervals were pro-rated based on the time remaining until sunset on the day of application. Following the 6-12 hour interval, sampling was completed on a schedule consistent with morning (after sunrise) and evening (prior to sunset) sampling times. The start and end times for all the post-application air sampling events are summarized in Appendix E. In-field

samples were stored in freezers while on site and shipped in coolers containing dry ice to the analytical facility.

To sample air moving off plot, eight perimeter air monitoring stations were located 1.5 m above the crop canopy and 5 m outside of the edge of the test substance treatment area (Figure 10). Air samplers at the eight perimeter sampling locations were turned on just after application. PUF collectors were collected from the perimeter stations at approximate nominal intervals of 6, 12, 24, 36, 48, 60, 72, 84, 96, 108, 120, 132, 144, 156, and 168 hours following completion of the application to the entire plot. Sample collections for the 0-6 hour and 6-12 hour intervals were pro-rated based on the time remaining until sunset on the day of application. Following the 6-12 hour interval, sampling was completed on a schedule consistent with morning (after sunrise) and evening (prior to sunset) sampling times. The start and end times for the post-application perimeter air sampling events are summarized in Appendix E. Perimeter samples were stored in freezers while on site and shipped in coolers containing dry ice to the analytical facility.

During post-application sample collection and installation of new PUF collector media, the air sampling pumps continued to run. Before PUF collector samples were collected, the air flow rate was measured using an SKC Check-Mate flow meter and recorded on a field form. After samples were collected and new PUF collectors were installed, the air sampling pumps were recalibrated to a flow rate of approximately 3.0 L/min using the same flow meter. The serial number of the flow meter used for calibration was recorded on each sample collection field form. Sample dates and times are provided in Appendix E.

## 2.15 Spray Drift Samples

Spray drift deposition transects were established perpendicular to the application area in all four directions (9 total transects; see Figure 8) with the filter papers placed at the following approximate distances from edge of the application area: 3, 5, 10, 20, 40, 50, 60 m. An additional filter paper sample was placed approximately 90 m from the application area for the four transects anticipated to lie downwind of the application area during the time of application. These transects included DWA, DWB, DWC, and LWA.

Sample collectors were placed horizontally at canopy height with Whatman #1, 15 cm diameter filter papers. The sample collectors consisted of a plexiglass platform that at each sampling event had a piece of cardboard was placed on top of to ensure a clean area to place to set the filter paper.

Filter papers were collected from each transect and distance at approximately 1, 24, 48, 72, 96, 120, 144, and 168 hours (8 periods) following initiation of the application to the application area. The start and end times for the spray drift deposition sampling events are summarized in Appendix E.

Spray drift sample collection was performed by LRC staff. Filter papers were collected by starting with the furthest distance samples and working toward the application area to reduce the potential for cross contamination. Nitrile gloves were worn in the collection of all filter paper samples. Gloves were replaced with new gloves prior to starting collection of a new transect. Each deposition filter paper was placed into pre-labeled 50 mL Falcon® polypropylene conical tube. Samples were transferred to an on-site freezer as soon as possible following collection. After rainfall events some filter papers were destroyed from the rainfall and unable to be collected.

## **2.16 Quality Control Samples**

Quality control samples utilized in the field phase of the study included field exposure and transit stability samples which were shipped from Eurofins (PUFs) and Monsanto Company (filter papers) overnight on dry ice under chain of custody to the field site in Shattuc, IL.

### **2.16.1 Field Exposed Spikes (PUF Collectors)**

Field exposed spikes were spiked in the lab at Eurofins and shipped overnight on dry ice to the field site. Field exposed spikes (spiked PUF collectors) were weathered for approximately 6 and 12 hours to determine the amount, if any, of dicamba that is lost during a sampling event. Weathering consisted of drawing air through the spiked PUF collectors using the same types of air sampling pumps set to the same flow rate used for air sampling described in Section 2.14. Weathering for the 6-hour spike samples began in the afternoon on July 7, 2019 and concluded that evening. Weathering for the 12-hour spike samples began in the afternoon on July 6, 2019 and concluded the next morning (July 7, 2019). The spike sampling was performed at the hotel that LRC sampling personnel stayed at in Mount Vernon, IL (approximately 26 miles south of the test site), where no additional exposure to test materials was expected to occur. For both time intervals, there were three replicates of PUFs fortified at four concentrations: 0, 3, 10, and 30 ng dicamba/PUF. Total there were 24 field exposed spike PUF samples.

### **2.16.2 Transit Stability (PUF Collectors and Filter Paper)**

A set of PUF collectors and a set of filter papers were prepared at Eurofins and Monsanto, respectively to evaluate stability of dicamba during transport.

Three replicates of PUF collectors were fortified at two concentrations: 0 and 30 ng dicamba/PUF. PUF collector fortifications occurred in the laboratory in Columbia, MO. Additionally, five replicates of filter paper were fortified at two concentrations: 0 and 0.05 µg dicamba/filter paper. Filter paper fortifications occurred in the laboratory in Chesterfield, MO. The samples were shipped on dry ice by overnight courier under chain of custody from Eurofins and Monsanto to the field site in Shattuc, IL. Upon receipt (July 02, 2019 for the filter papers and July 03, 2019 for the PUF collectors), the samples were stored at approximately -20 °C.

Chain of custody documents were completed, and the samples were shipped on dry ice by LRC staff under chain of custody to the laboratories.

## **2.17 Crop Destruct**

The study protocol did not require crop destruction.

## **2.18 Control of Bias**

Bias and cross-contamination was controlled by thoroughly cleaning spray equipment prior to application and with detailed procedures for obtaining representative samples while avoiding contamination.

Separate sample crews collected the downwind and upwind deposition samples to reduce cross contamination. Deposition samples were collected starting with the furthest downwind samples and working toward the application area. Nitrile protective gloves were changed prior to commencing the collection of the next transect at the furthest downwind sample location.

The center mast sample team used appropriate PPE for entering the application area and changed protective gloves in-between collecting PUF samples and deploying new PUF samples. Sample collection started with the highest collector on the center mast and worked to the lowest.

Field volatility tarps were removed carefully in such a way that minimized contamination and damage to the tarped soybeans. Tarps were removed starting with the edge furthest from the application area and working toward the application area.

## **2.19 Deviations from Protocol**

- 1- Soil sample for soil characterization was shipped chilled instead of ambient.
  - a. Protocol Section 6.7.1
  - b. Researcher oversight
- 2- Water holding capacity for 15 bar (disturbed) was not requested or performed for soil characterization
  - a. Protocol Section 6.7.1
  - b. Researcher oversight
- 3- Series 3 test was requested and performed for water characterization instead of series 5. The following parameters were not measured in a Series 3 but would have been measured in a Series 5 test: potassium, carbonate, bicarbonate, nitrate, sulfate, chloride, and alkalinity. The following parameters were not measured in a Series 5 but were measured in a Series 3 test: total dissolved solids, and turbidity.
  - a. Protocol Section 6.7.1
  - b. Researcher oversight
- 4- The tractor sprayer speed was not calibrated 3 times prior to application.
  - a. Protocol section 6.6

- b. Researcher oversight
- 5- A total of 47 deposition filter paper samples were unable to be collected due to effects from rain. The samples unable to be collected are listed in Protocol Change Document No. 4.
  - a. Protocol Section 6.7.4
  - b. This deviation expected to have minimum negative effect on the study.

### 3.0 RESULTS

#### 3.1 Test Substance Spray Rate Verification and Spray Application

During the sprayer/nozzle verification, the measured average flow rate was 1.94 L/min (0.512 gal/min) for the TTI 11004 nozzles (Table 6). The total calculated boom output based on the measured nozzle flow rates was 186.71 L/min (49.32 gal/min).

The target sprayer speed was 13.6 mph however, due to the required path to be driven by the sprayer, during passes 1 through 4 the sprayer speed was unable to be consistent across the entire pass. Variable flow rate technology was used to compensate the sprayer output based on speed to keep the output at 15 GPA. For passes 5-10, where the sprayer was able to keep a consistent speed across the entire pass length, the average pass time was 102.8% of target. The percent of target for all pass times (1-10), was 102.7%. Sprayer pass times presented in Table 6.

#### 3.2 Test Site Environmental Monitoring (During and After Application)

The application to the dicamba-tolerant soybean plot occurred from 9:35 to 9:49 am on July 2, 2019.

As recorded by the site 10-meter main meteorological station at the 1.7 m height:

- the average wind speed of the one-minute averages during the treatment application was 4.88 mph (2.182 m/s). The mean wind direction during application of the one-minute averages was 235.1°. The target wind direction was 225°. The wind speed ranged from a minimum of 1.21 mph (0.540 m/s) to a maximum of 10.58 mph (4.730 m/s) during application (Table 4).
- The maximum temperature and relative humidity recorded during application to the test plot were 85.44°F (29.69°C) and 75.39%, respectively (Table 3).
- During the first post-application sampling period (0-6 hours), the maximum temperature was 92.08°F (33.38°C) (Table 5).
- For the entire Treatment 1 post-application air sampling period (10:10 am on July 2, 2019 to 7:39 am on July 9, 2019), the maximum, and minimum air temperatures were 92.80°F (33.78°C) and 67.15°F (19.53°C), respectively (Table 5). The lowest temperature



recorded during the Treatment 1 post-application air sampling period occurred during the 132-144-hour sampling period.

- A total of 2.35 in (59.69 mm) of rainfall was recorded during the air sampling portion of the study. Totals of 0.07, 2.01, 0.13 and 0.14 inches of rain fall was recorded during the 0-6, 36-48, 84-96, and 96-108 hour sampling events respectively.

The daily average, daily minimum, and daily maximum temperatures for each day of the study are presented in Table 5. Meteorological electronic raw data are archived with the study records.

### **3.3 Test Plot Soil and Water Characterization**

AGVISE Laboratories received the soil characterization samples on July 11, 2019 and reported the results on July 17, 2019. The USDA textural class of the soil samples was determined to be silt loam using the hydrometer method (13% sand, 66% silt, and 21% clay). Table 1 provides the complete soil characterization performed by AGVISE Laboratories (Appendix D).

AGVISE Laboratories received the water characterization samples on July 11, 2019 and reported the results on July 17, 2019. AGVISE Laboratories determined that the water sample had a pH of 7.8. Table 2 provides the complete water characterization report by AGVISE Laboratories (Appendix D).

### **3.4 Tank Mix Samples**

Summary data for tank mix analysis are presented in the Eurofins Analytical Sub-Report (an Appendix of the Main Report).

### **3.5 Application Verification Filter Paper Samples**

Summary data for application verification sample analysis are presented in the Eurofins Analytical Sub-Report (an Appendix of the Main Report).

### **3.6 Flux Monitoring Samples (Field Volatility)**

#### **3.6.1 Pre-Application Samples**

For information on the pre-application PUF collectors, refer to the Volatile Flux Estimation Sub-Report and Eurofins Analytical Sub-Report (Appendices to the Main Report).

#### **3.6.2 Post-Application Samples**

For information on the post application PUF collectors, refer to the Volatile Flux Estimation Sub-Report and Eurofins Analytical Sub-Report (Appendices to the Main Report).

### **3.6.3 Transit Stability Air Samples**

To mimic sample transportation from the field to Eurofins, three PUF samples were fortified at Eurofins at 30 ng dicamba/PUF and placed on dry ice along with three untreated control PUF samples. These samples were designated as transit stability samples and their analytical recovery values are presented in the Eurofins Analytical Sub-Report (an Appendix to the Main Report).

### **3.6.4 Field Exposed Spikes**

There were three field exposed spike samples at each of the following concentration levels: 0, 3, 10, and 30 ng dicamba/PUF. The samples were shipped on dry ice to the field site where they weathered for approximately 6 and 12 hours. These samples were designated as field spike samples and their analytical recovery values are presented in the Eurofins Analytical Sub-Report (an Appendix to the Main Report).

## **3.7 Spray Drift Deposition Samples**

For information on the Drift Deposition samples, refer to the Spray Drift Deposition Modeling Sub-Report and Monsanto Analytical Sub-Report (Appendices to the Main Report).

### **3.7.1 Transit Stability Filter Paper Samples**

To mimic sample transportation from the field to Monsanto, five replicates of filter papers were fortified in the Monsanto lab at 0.05 µg dicamba/filter paper and placed on dry ice along with five untreated control filter papers. These samples were designated as transit stability spray drift samples and their analytical recovery values are presented in the Monsanto Analytical Sub-Report (an Appendix to the Main Report).

## **3.8 Plant Effects Samples**

Plant effects were assessed for plant heights and visual symptomology for nominal 0, 14, and 28 Days After Treatment (DAT) around the test plot (Figure 11). Additional information can be found in the Plant Effects (Drift and Volatilization) Sub-Report (an Appendix to the Main Report).

## **4.0 CONCLUSIONS**

A field study measuring volatilization, spray drift, and plant effects was performed in Shattuc, Clinton County, Illinois to measure flux of dicamba following spray applications of a mixture of MON 76980 at a rate of 0.50 lb dicamba a.e./A (0.56 kg dicamba a.e./ha) to a field planted with both dicamba-tolerant and dicamba non-tolerant emergent soybean. Based on the pass time and spray calibration results, and the application verification samples, the dicamba formulation was successfully applied near the target rate of 0.5 lb a.e./A, with individual spray swaths ranging from 97.7 % to 106.9 % of the target rate. It is also presumed that the use of variable rate

technology was able to adjust the total application rate closer to the target rate than what could be achieved based on calculated pass times and calibrated spray rate alone.

During the study, based on minute-averaged data from the 10-meter main meteorological station, the maximum measured air temperature at 1.7 m height was 92.80 °F (33.78 °C) for the test plot. The maximum measured surface soil temperature (1 mm depth) was 102 °F (38.89 °C) for the test plot.

## 5.0 REFERENCES

ASABE S561.1 (2009) Procedure for Measuring Drift Deposits from Ground, Orchard, and Aerial Sprayers. American Society of Agricultural and Biological Engineers, St. Joseph, MI.

ISO Standard 22866 (2005) Equipment for Crop Protection – Methods for Field Measurement of Spray Drift. International Standards Organization

US EPA (1998) Spray Drift Test Guidelines, OPPTS 840.1200 Spray Drift Field Deposition. United States Environmental Protection Agency, Prevention, Pesticides, and Toxic Substances. EPA 712-C-98-112.

US EPA. (2008) AERSURFACE User's Guide, United States Environmental Protection Agency, Office of Air Quality Planning and Standards, Emissions, Monitoring, and Analysis Division. Research Triangle Park, North Carolina, EPA-454/B-08-001.

## **6.0 TABLES**

**Table 1. AGVISE Soil Characteristics**

<b>Soil Characterization Parameter<sup>1</sup></b>	<b>Results</b>	
Percent Sand	13	
Percent Silt	66	
Percent Clay	21	
USDA Textural Class (hydrometer method)	Silt Loam	
Bulk Density (disturbed) gm/cc	1.14	
Cation Exchange Capacity (meq/100 g)	9.8	
% Moisture at 1/3 Bar	28.3	
% Organic Matter – Walkley Black	1.8	
pH in 1:1 soil:water ratio	6.8	
Base Saturation – Calcium	67.9%	1330 ppm
Base Saturation – Magnesium	8.9%	105 ppm
Base Saturation – Sodium	1.1%	25 ppm
Base Saturation – Potassium	3.3%	127 ppm
Base Saturation – Hydrogen	18.8%	18 ppm

<sup>1</sup>Note: Data for 0-6” depth composited soil samples

**Table 2. AGVISE Water Characteristics**

Water Characterization Parameter	Results
pH	7.8
Calcium	29 ppm
Magnesium	11 ppm
Sodium	16 ppm
Hardness	119 mg equivalent CaCO <sub>3</sub> /L
Conductivity	0.33 mmhos/cm
Sodium Adsorption Ratio (SAR)	0.65

**Table 3. Meteorological Conditions During Spray Application**

Meteorological Condition <sup>1</sup>	Treatment
Application Date	02-July-2019
Time Range	9:35 am – 9:49 am
Wind Speed Range (meters/second)*	0.54 – 4.73
Average Wind Speed (meters/second)*	2.182
Average Wind Direction (Degrees)*	235.1
Maximum Temperature (°C)*	29.69
Maximum Relative Humidity (%)*	75.39
Maximum Solar Radiation (kW/m <sup>2</sup> )*	0.712
Maximum Soil Moisture, 2" (m <sup>3</sup> /m <sup>3</sup> )	0.366
Maximum Soil Temperature, Surface (1mm) (°C)	30.69
Maximum Soil Temperature, 2" (°C)	27.23
Maximum Soil Temperature, 6" (°C)	24.79

<sup>1</sup> 10-meter main meteorological station.

\* Data from the 1.7 m height.



**Table 4. Period Statistics for Wind Data from Main Meteorological Station**

Sampling Period	Start <sup>1</sup>	End <sup>2</sup>	Average Wind Speed (m/s) at Heights (m)			Average Wind Direction (°) at Heights (m)		
			1.7	5	10	1.7	5	10
0-6	7/2/19 10:10 AM	7/2/19 3:29 PM	2.636	3.113	3.519	225.5	222.2	223.5
6-12	7/2/19 3:33 PM	7/2/19 6:04 PM	2.081	2.494	2.791	199.1	198.8	205.0
12-24	7/2/19 6:09 PM	7/3/19 7:54 AM	1.497	1.912	2.403	190.8	187.5	186.9
24-36	7/3/19 7:58 AM	7/3/19 4:45 PM	1.902	2.157	2.385	252.2	246.9	247.7
36-48	7/3/19 4:51 PM	7/4/19 7:46 AM	2.050	2.497	2.924	140.7	135.1	134.8
48-60	7/4/19 7:50 AM	7/4/19 5:34 PM	2.797	3.247	3.677	189.1	184.9	183.9
60-72	7/4/19 5:38 PM	7/5/19 7:46 AM	2.090	2.578	3.071	156.8	152.4	152.6
72-84	7/5/19 7:48 AM	7/5/19 4:34 PM	2.787	3.304	3.674	178.7	179.4	179.1
84-96	7/5/19 4:37 PM	7/6/19 8:01 AM	1.521	1.806	2.126	221.3	215.0	215.3
96-108	7/6/19 8:04 AM	7/6/19 6:09 PM	2.649	3.134	3.522	285.4	280.1	279.9
108-120	7/6/19 6:12 PM	7/7/19 7:42 AM	1.544	2.020	2.475	198.4	205.5	205.3
120-132	7/7/19 7:45 AM	7/7/19 6:13 PM	2.585	3.050	3.225	218.6	236.9	241.1
132-144	7/7/19 6:18 PM	7/8/19 7:48 AM	1.663	2.121	2.323	144.0	204.7	214.1
144-156	7/8/19 7:50 AM	7/8/19 6:02 PM	2.333	2.716	3.042	64.9	62.5	64.3
156-168	7/8/19 6:06 PM	7/9/19 7:39 AM	1.445	2.010	2.720	83.1	79.6	82.3

<sup>1</sup> Start time = time the first sample on the center mast was started.

<sup>2</sup> End time = time the last sample on the center mast was collected.

**Table 5. Period Statistics for Temperature and Relative Humidity Data from Main Meteorological Station**

Sampling Period (hr)	Start	End	Average Temperature (°C) at Heights (m)			Absolute Maximum Temperature (°C) at Heights (m)			Absolute Minimum Temperature (°C) at Heights (m)			Minimum Relative Humidity (%) at Heights (m)	Maximum Relative Humidity (%) at Heights (m)
			1.7	5	10	1.7	5	10	1.7	5	10	1.7	1.7
0-6	7/2/19 10:10 AM	7/2/19 3:29 PM	30.16	30.06	29.65	33.38	33.12	32.32	24.71	24.99	24.91	46.28	86.50
6-12	7/2/19 3:33 PM	7/2/19 6:04 PM	30.44	30.56	30.29	32.75	32.40	32.35	27.79	28.08	27.69	51.66	78.73
12-24	7/2/19 6:09 PM	7/3/19 7:54 AM	25.58	25.99	25.83	32.06	32.28	31.35	22.26	22.55	22.30	57.08	97.30
24-36	7/3/19 7:58 AM	7/3/19 4:45 PM	30.46	30.29	30.44	33.78	33.48	33.66	25.42	25.45	25.67	46.03	86.90
36-48	7/3/19 4:51 PM	7/4/19 7:46 AM	22.25	22.45	22.09	33.67	33.50	32.96	20.42	20.68	19.22	47.02	100.00
48-60	7/4/19 7:50 AM	7/4/19 5:34 PM	28.90	28.80	28.51	32.49	32.50	32.03	23.52	23.26	22.59	57.14	91.00
60-72	7/4/19 5:38 PM	7/5/19 7:46 AM	25.56	25.83	25.54	29.32	29.39	28.71	23.09	23.42	23.01	67.57	97.40
72-84	7/5/19 7:48 AM	7/5/19 4:34 PM	29.21	29.07	28.59	33.46	32.96	32.06	26.02	26.01	24.95	55.64	89.40
84-96	7/5/19 4:37 PM	7/6/19 8:01 AM	24.30	24.53	24.18	26.69	26.84	26.57	22.85	23.12	22.63	80.10	100.00
96-108	7/6/19 8:04 AM	7/6/19 6:09 PM	28.84	28.66	28.64	33.36	33.01	32.95	23.99	24.12	23.35	52.54	96.10
108-120	7/6/19 6:12 PM	7/7/19 7:42 AM	25.93	26.34	26.12	30.57	30.80	30.54	22.53	22.58	22.14	70.48	98.50
120-132	7/7/19 7:45 AM	7/7/19 6:13 PM	28.36	28.09	27.59	31.67	31.16	30.25	23.15	22.81	22.31	54.25	86.90
132-144	7/7/19 6:18 PM	7/8/19 7:48 AM	23.52	23.89	23.68	30.54	30.39	29.61	19.53	19.93	19.34	58.20	95.40
144-156	7/8/19 7:50 AM	7/8/19 6:02 PM	28.08	27.85	27.33	31.51	31.16	30.57	21.77	21.72	21.04	51.48	85.20
156-168	7/8/19 6:06 PM	7/9/19 7:39 AM	24.10	24.64	24.65	30.71	30.73	30.29	20.68	21.32	20.99	54.04	96.80

<sup>1</sup> Start time = time the first sample on the center mast was started.<sup>2</sup> End time = time the last sample on the center mast was collected.

**Table 6. Spray Application Verification Information**

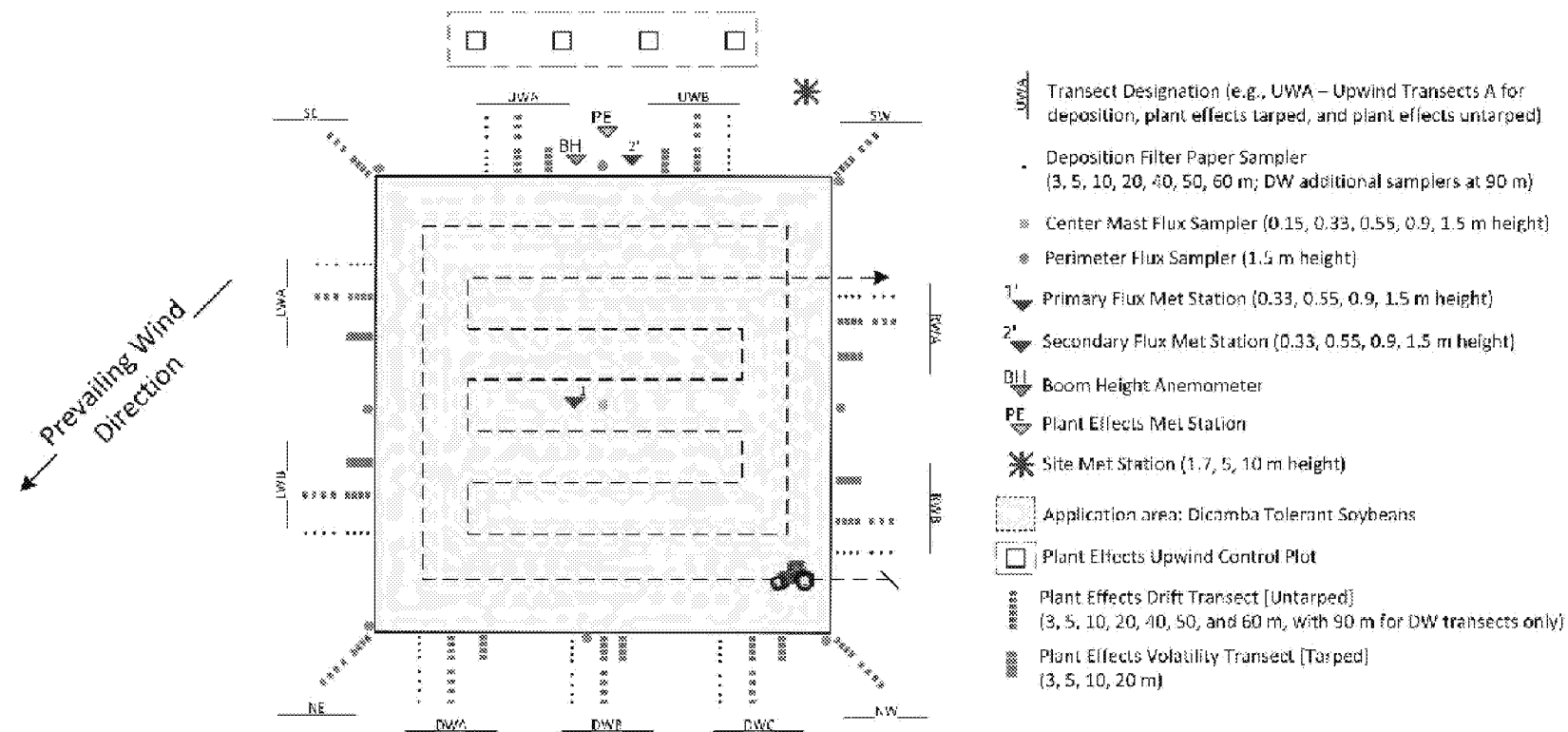
<b>Pass Number</b>				
	<b>Application Pass Time (sec)</b>	<b>Pass Length (ft)</b>	<b>Target Pass Time (sec)</b>	<b>% of Target</b>
1	41.64	850	42.61	97.7
2	43.32	840	42.11	102.9
3	43.88	850	42.61	103.0
4	45.02	840	42.11	106.9
5	36.89	730	36.60	100.8
6	37.23	730	36.60	101.7
7	37.56	730	36.60	102.6
8	37.81	730	36.60	103.3
9	38.06	730	36.60	104.0
10	38.16	730	36.60	104.3
<b>Percent applied based on passes 5-10</b>				<b>102.8</b>
<b>Percent applied based on all passes</b>				<b>102.7</b>

## **7.0 FIGURES**

**Figure 1. Study Site**

Aerial photo showing the treated area (outlined in orange) looking north. The covered transects outside of the treated area can also be seen.

### Figure 2. Test Plot Diagram



\* Figure not drawn to scale.

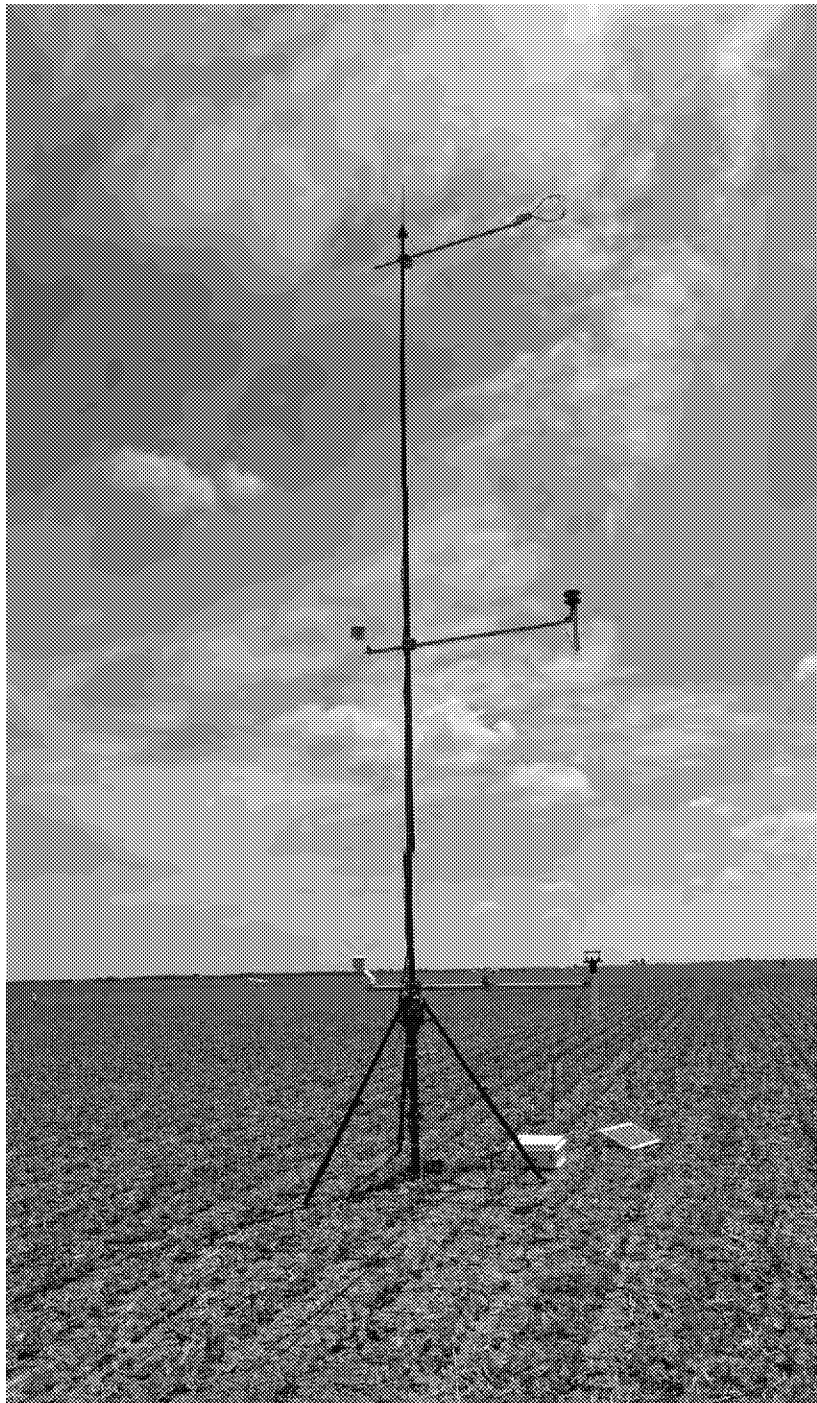
**Figure 3. Test Site Location Relative to the Field Cooperator's Shop in Shattuc, IL**

**Figure 4. Ground Boom Sprayer**





**Figure 5. 10-Meter Main Meteorological Station**



**Figure 6. Boom Height Anemometer**



**Figure 7. Flux Meteorological Station**



**Figure 8. Spray Drift Deposition Sampling Platform**





**Figure 9. In-Field PUF Sampling Station**



**Figure 10. Perimeter PUF Sampling Station**



**Figure 11. Plant Effects-Volatilization Sampling Area**

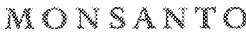

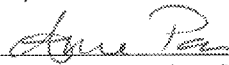


Photo taken 28 DAT

## **8.0 APPENDICES**



## **Appendix A. Certificate of Analysis MON 76980 and MON 79789**

 		<b>Certificate of Analysis</b>		<b>Testing Facility:</b> Monsanto 800 N. Lindbergh Blvd St. Louis, MO 63137 314.694.1000
Sample ID:		MON 76980 Lot: 11495284		
Lot Number		11495284		
Storage Conditions:		Warehouse (35 to 100°F)		
<b>Tests Performed:</b> <input checked="" type="checkbox"/> <b>Appearance:</b> Blue Liquid <input type="checkbox"/> <b>Identity confirmation (for TGA or PAI):</b> <input checked="" type="checkbox"/> <b>Active Ingredient Assay</b>				
Ingredient		Assay Method		Result
Dicamba		ME-1699		28.65% wt. (a.e.) 343 g/L
<input checked="" type="checkbox"/> <b>Other Tests</b>				
Test Required		Test Method		Result
Density		EQ-0982		1.1971 g/mL
Analysis Date:		10-May-19		
Expiration Date:		10-May-20		<input type="checkbox"/> Expiration date unknown
If unknown Expiration Date, enter the Reanalysis Date: <a href="#">Click here to choose the date.</a>				
<b>GLP Statement:</b> Data generated in the certification of this material is in compliance with Good Laboratory Practices (40 CFR § 160), with the following exceptions:  <input checked="" type="checkbox"/> No GLP Exceptions.				
Comments:				
Issued by:  Anne Park				
Date: <u>6 June 2019</u> Date				

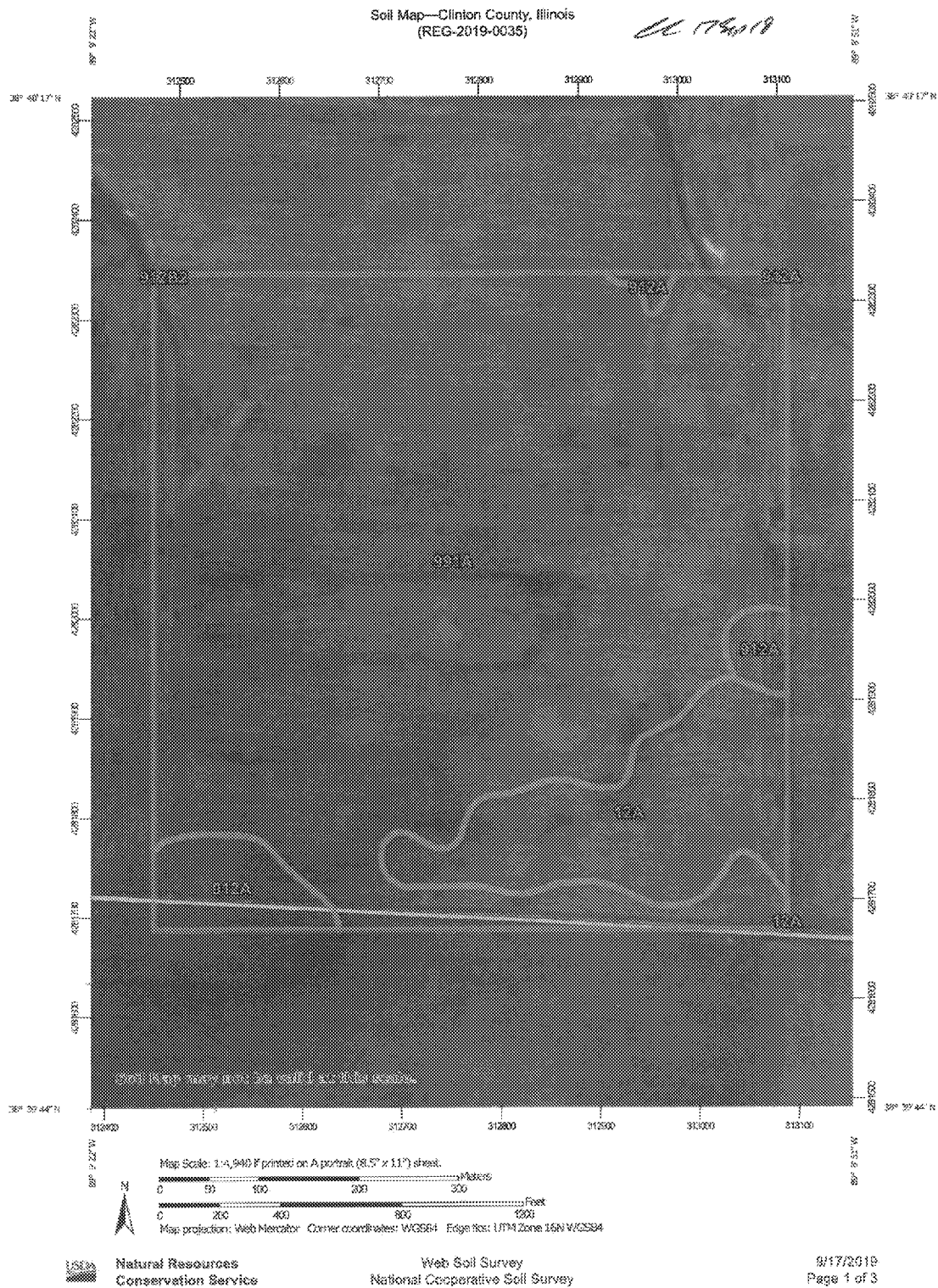
		<b>Certificate of Analysis</b>		<b>Testing Facility:</b> Monsanto 800 N. Lindbergh Blvd St. Louis, MO 63137 314.694.1000
Sample ID:		MON 79789, Lot # 11495283		
Lot Number		11495283		
Storage Conditions:		Warehouse (35 to 100°F)		
<b>Tests Performed:</b>				
<input checked="" type="checkbox"/> <b>Appearance:</b> Light brown, clear, viscous liquid				
<input type="checkbox"/> <b>Identity confirmation (for TGA1 or PA1):</b>				
<input checked="" type="checkbox"/> <b>Active Ingredient Assay</b>				
Ingredient		Assay Method		Result
Glyphosate		HPLC-RI		39.74 % wt. (a.e.), 538 g/L
<input checked="" type="checkbox"/> <b>Other Tests</b>				
Test Required		Test Method		Result
Density		EQ-0982		1.3526 g/mL
Analysis Date:		7-May-19		
Expiration Date:		7-May-20		<input type="checkbox"/> Expiration date unknown
If unknown Expiration Date, enter the Reanalysis Date: <a href="#">Click here to choose the date.</a>				
<b>GLP Statement:</b> Data generated in the certification of this material is in compliance with Good Laboratory Practices (40 CFR § 160), with the following exceptions:				
<input checked="" type="checkbox"/> No GLP Exceptions.				
Comments:				
Issued by: 				
Henry Valentin		05/21/2019 Date		

**Appendix B. Agronomic and Pesticide Use History**

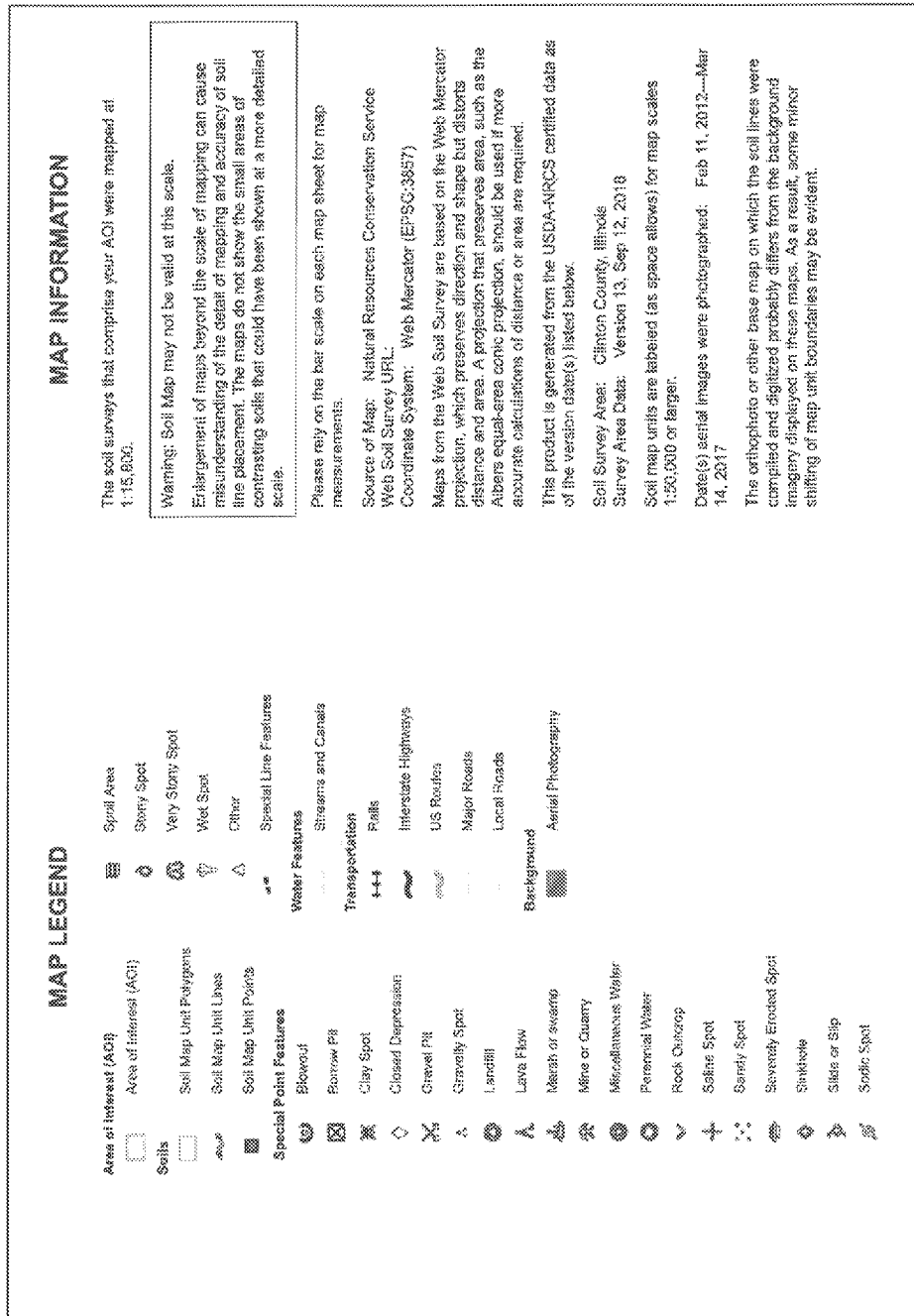
<b>Pesticide History</b>				
<b>Year</b>	<b>Crop</b>	<b>Pesticide</b>	<b>Date</b>	<b>Rate</b>
2019	Soybean	Gramoxone XL	4-Jun-2019	3 pt/A
		Intimidator	4-Jun-2019	2.75 pt/A
		LI700	4-Jun-2019	4 fl oz/A
2018	Corn	AMS	5-Jun-2018	1.5 lb/A
		FS MicroNut.	5-Jun-2018	1 qt/A
		Roundup Powermax	5-Jun-2018	36 fl oz/A
		Degree Extra	16-May-2018	3.25 qt/A
		LAMCAP II	16-May-2018	1.9 fl oz/A
		Roundup Powermax	16-May-2018	32 fl oz/A
		32% w/Sulfur Blend	7-May-2018	10.72 gal/A
		28-0-0-12	7-May-2018	32 fl oz/A
		Anhydrous Ammonia	27-Apr-2018	188.05 lb/A
		Drexel Simazine 4L	26-Apr-2018	1.5 qt/A
		Roundup Powermax	26-Apr-2018	32 fl oz/A
		Salvo	26-Apr-2018	10 fl oz/A
2017*	Soybean	Roundup Powermax	2017	32 oz/A
		Salvo 2,4-D	2017	12 fl oz/A
		Authority XL	2017	4 fl oz/A
		Warrant	2017	2.5 pt/A
		Xtendimax	2017	22 fl oz/A
		Roundup Powermax	2017	30 fl oz/A
		Intact	2017	Unknown
2016	Corn	Degree Extra	31-May-2016	3.25 qt/A
		Roundup Powermax	31-May-2016	32 fl oz/A
		Salvo 2,4-D	31-May-2016	7.5 fl oz/A
		Tombstone	31-May-2016	2 fl oz/A
		Gramoxone	25-May-2016	3.25 qt/A
		Tombstone	25-May-2016	2 fl oz/A
		Anhydrous Ammonia	16-Apr-2016	174.72 gal/A
		Roundup Powermax	5-Apr-2016	30 fl oz/A
		Salvo 2,4-D	5-Apr-2016	10 fl oz/A
		Simazine 90DF	5-Apr-2016	1 lb/A
		Anhydrous Ammonia	5-Apr-2016	161.92 gal/A

\*Exact dates unknown for 2017 applications.

## **Appendix C. USDA NRCS Clinton County, IL Soil Report**



Soil Map—Clinton County, Illinois  
(REG-2019-0035)



SR172019  
Page 2 of 3

Web Soil Survey  
National Cooperative Soil Survey

USDA  
Natural Resources  
Conservation Service

Soil Map—Clinton County, Illinois

REG-2019-0035

### Map Unit Legend

Map Unit Symbol	Map Unit Name	Acres in AOI	Percent of AOI
12A	Wynoose silt loam, 0 to 2 percent slopes	12.2	11.6%
912A	Hoyleton-Darmstadt silt loams, 0 to 2 percent slopes	5.0	4.8%
912B2	Hoyleton-Darmstadt silt loams, 2 to 5 percent slopes, eroded	0.0	0.0%
* 991A	Cisne-Huey silt loams, 0 to 2 percent slopes	86.9	83.4%
<b>Totals for Area of Interest</b>		<b>104.1</b>	<b>100.0%</b>

USDA  
Natural Resources  
Conservation Service

Web Soil Survey  
National Cooperative Soil Survey

9/17/2019  
Page 3 of 3



## **Appendix D. AGVISE Soil and Water Characterization Reports**



604 Highway 15 West  
P.O. Box 516  
Northwood, ND 58267  
(701) 587-6010  
FAX (701) 587-6013  
email: agvise@polar.com  
Homepage: www.agvise.com

## AGVISE Soil Characterization Report

Submitting firm = LANGE RESEARCH  
Protocol or Study No = REG-2019-0035  
Sample ID. = REG-2019-0035-SOILCHAR  
Sample Depth = 0-6"  
Trial ID. = NA  
Date Received: 7/11/19 Date Reported: 07-17-2019

AGVISE Lab No: 19-1371 Acct No: LA6122

Percent Sand 13  
Percent Silt 66  
Percent Clay 21  
USDA Textural Class (hydrometer method) Silt Loam


Bulk Density (disturbed) gm/cc 1.14  
Cation Exchange Capacity (meq/100 g) 9.8

% Moisture at 1/3 Bar 28.3  
% Organic Matter--Walkley Black 1.8  
pH in 1:1 soil:water ratio 6.8

## Base Saturation Data

Cation	Percent	ppm
Calcium	67.9	1330
Magnesium	8.9	105
Sodium	1.1	25
Potassium	3.3	127
Hydrogen	18.8	18

These tests were completed in compliance of 40 CFR Part 160.

  
Larry Wikoff  
Analytical Investigator

Date

7/17/19

REG-2019-0035

 23 Jul 19

Agricultural Testing



REG-2019-0035  
06/22/19

604 Highway 15 West  
P.O. Box 510  
Northwood, ND 58267  
(701) 587-6010  
FAX (701) 587-6013  
email: [agvise@polarcenter.com](mailto:agvise@polarcenter.com)  
Homepage: [www.agvise.com](http://www.agvise.com)

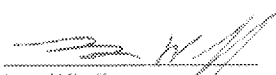
### AGVISE GLP Water Characterization Report

Submitting Account = LA6122  
Submitting Firm = LANGE RESEARCH  
Protocol or Study # = REG-2019-0035  
Sample ID = REG-2019-0035-WATERANALY  
Trial ID = NA  
Date Received = 07-11-2019  
Date Reported = 07-17-2019

AGVISE Lab # 19-115

pH	7.8
Calcium	29 ppm
Magnesium	11 ppm
Sodium	16 ppm
Hardness	119 mg equivalent CaCO <sub>3</sub> /L
Conductivity	0.33 mhos/cm
Sodium Adsorption Ratio (SAR)	0.65
Total Dissolved Solids	180 ppm
Turbidity	13.1 NTU

These tests were completed in compliance of 40 CFR Part 160.

  
Larry Wikoff  
Nutrient Laboratory Director

7/12/19  
Date

----- Agricultural Testing -----

**Appendix E. Sample Period Collection Dates and Times**

<b>Sample Description</b>	<b>Height (cm) or Location</b>	<b>Sample Period</b>	<b>Rep</b>	<b>Sample Start Date</b>	<b>Sample Start Time</b>	<b>Sample End Date</b>	<b>Sample End Time</b>
Center Mast	15	PRE	1	6/30/2019	1114	6/30/2019	1716
Center Mast	15	PRE	2	6/30/2019	1116	6/30/2019	1717
Center Mast	15	0-6 hr	1	7/2/2019	1010	7/2/2019	1529
Center Mast	33	0-6 hr	1	7/2/2019	1010	7/2/2019	1527
Center Mast	55	0-6 hr	1	7/2/2019	1010	7/2/2019	1525
Center Mast	90	0-6 hr	1	7/2/2019	1010	7/2/2019	1524
Center Mast	150	0-6 hr	1	7/2/2019	1010	7/2/2019	1521
Center Mast	150	0-6 hr	2	7/2/2019	1010	7/2/2019	1522
Center Mast	15	6-12 hr	1	7/2/2019	1543	7/2/2019	1804
Center Mast	33	6-12 hr	1	7/2/2019	1541	7/2/2019	1803
Center Mast	55	6-12 hr	1	7/2/2019	1538	7/2/2019	1801
Center Mast	90	6-12 hr	1	7/2/2019	1536	7/2/2019	1800
Center Mast	150	6-12 hr	1	7/2/2019	1533	7/2/2019	1757
Center Mast	150	6-12 hr	2	7/2/2019	1535	7/2/2019	1758
Center Mast	15	12-24 hr	1	7/2/2019	1820	7/3/2019	0754
Center Mast	33	12-24 hr	1	7/2/2019	1816	7/3/2019	0752
Center Mast	55	12-24 hr	1	7/2/2019	1815	7/3/2019	0751
Center Mast	90	12-24 hr	1	7/2/2019	1814	7/3/2019	0750
Center Mast	150	12-24 hr	1	7/2/2019	1809	7/3/2019	0748
Center Mast	150	12-24 hr	2	7/2/2019	1812	7/3/2019	0750
Center Mast	15	24-36 hr	1	7/3/2019	0807	7/3/2019	1645
Center Mast	33	24-36 hr	1	7/3/2019	0806	7/3/2019	1644
Center Mast	55	24-36 hr	1	7/3/2019	0805	7/3/2019	1644
Center Mast	90	24-36 hr	1	7/3/2019	0803	7/3/2019	1643
Center Mast	150	24-36 hr	1	7/3/2019	0758	7/3/2019	1641
Center Mast	150	24-36 hr	2	7/3/2019	0759	7/3/2019	1642
Center Mast	15	36-48 hr	1	7/3/2019	1655	7/4/2019	0746
Center Mast	33	36-48 hr	1	7/3/2019	1654	7/4/2019	0744
Center Mast	55	36-48 hr	1	7/3/2019	1653	7/4/2019	0743
Center Mast	90	36-48 hr	1	7/3/2019	1652	7/4/2019	0742
Center Mast	150	36-48 hr	1	7/3/2019	1651	7/4/2019	0739
Center Mast	150	36-48 hr	2	7/3/2019	1651	7/4/2019	0740

**Appendix E. Sample Period Collection Dates and Times (continued)**

<b>Sample Description</b>	<b>Height (cm) or Location</b>	<b>Sample Period</b>	<b>Rep</b>	<b>Sample Start Date</b>	<b>Sample Start Time</b>	<b>Sample End Date</b>	<b>Sample End Time</b>
Center Mast	15	48-60 hr	1	7/4/2019	0759	7/4/2019	1734
Center Mast	33	48-60 hr	1	7/4/2019	0758	7/4/2019	1733
Center Mast	55	48-60 hr	1	7/4/2019	0755	7/4/2019	1732
Center Mast	90	48-60 hr	1	7/4/2019	0753	7/4/2019	1731
Center Mast	150	48-60 hr	1	7/4/2019	0750	7/4/2019	1729
Center Mast	150	48-60 hr	2	7/4/2019	0752	7/4/2019	1730
Center Mast	15	60-72 hr	1	7/4/2019	1745	7/5/2019	0746
Center Mast	33	60-72 hr	1	7/4/2019	1743	7/5/2019	0745
Center Mast	55	60-72 hr	1	7/4/2019	1742	7/5/2019	0744
Center Mast	90	60-72 hr	1	7/4/2019	1740	7/5/2019	0744
Center Mast	150	60-72 hr	1	7/4/2019	1738	7/5/2019	0742
Center Mast	150	60-72 hr	2	7/4/2019	1739	7/5/2019	0743
Center Mast	15	72-84 hr	1	7/5/2019	0755	7/5/2019	1634
Center Mast	33	72-84 hr	1	7/5/2019	0753	7/5/2019	1633
Center Mast	55	72-84 hr	1	7/5/2019	0752	7/5/2019	1632
Center Mast	90	72-84 hr	1	7/5/2019	0750	7/5/2019	1631
Center Mast	150	72-84 hr	1	7/5/2019	0748	7/5/2019	1630
Center Mast	150	72-84 hr	2	7/5/2019	0749	7/5/2019	1629
Center Mast	15	84-96 hr	1	7/5/2019	1637	7/6/2019	0801
Center Mast	33	84-96 hr	1	7/5/2019	1642	7/6/2019	0800
Center Mast	55	84-96 hr	1	7/5/2019	1641	7/6/2019	0759
Center Mast	90	84-96 hr	1	7/5/2019	1639	7/6/2019	0758
Center Mast	150	84-96 hr	1	7/5/2019	1639	7/6/2019	0756
Center Mast	150	84-96 hr	2	7/5/2019	1638	7/6/2019	0757
Center Mast	15	96-108 hr	1	7/6/2019	0812	7/6/2019	1809
Center Mast	33	96-108 hr	1	7/6/2019	0810	7/6/2019	1808
Center Mast	55	96-108 hr	1	7/6/2019	0808	7/6/2019	1807
Center Mast	90	96-108 hr	1	7/6/2019	0807	7/6/2019	1805
Center Mast	150	96-108 hr	1	7/6/2019	0804	7/6/2019	1804
Center Mast	150	96-108 hr	2	7/6/2019	0806	7/6/2019	1805
Center Mast	15	108-120 hr	1	7/6/2019	1819	7/7/2019	0742
Center Mast	33	108-120 hr	1	7/6/2019	1817	7/7/2019	0742
Center Mast	55	108-120 hr	1	7/6/2019	1816	7/7/2019	0741
Center Mast	90	108-120 hr	1	7/6/2019	1815	7/7/2019	0739
Center Mast	150	108-120 hr	1	7/6/2019	1812	7/7/2019	0738
Center Mast	150	108-120 hr	2	7/6/2019	1814	7/7/2019	0738

**Appendix E. Sample Period Collection Dates and Times (continued)**

Sample Description	Height (cm) or Location	Sample Period	Rep	Sample Start Date	Sample Start Time	Sample End Date	Sample End Time
Center Mast	15	120-132 hr	1	7/7/2019	0751	7/7/2019	1813
Center Mast	33	120-132 hr	1	7/7/2019	0750	7/7/2019	1811
Center Mast	55	120-132 hr	1	7/7/2019	0748	7/7/2019	1810
Center Mast	90	120-132 hr	1	7/7/2019	0748	7/7/2019	1808
Center Mast	150	120-132 hr	1	7/7/2019	0745	7/7/2019	1806
Center Mast	150	120-132 hr	2	7/7/2019	0746	7/7/2019	1806
Center Mast	15	132-144 hr	1	7/7/2019	1825	7/8/2019	0748
Center Mast	33	132-144 hr	1	7/7/2019	1823	7/8/2019	0747
Center Mast	55	132-144 hr	1	7/7/2019	1821	7/8/2019	0746
Center Mast	90	132-144 hr	1	7/7/2019	1820	7/8/2019	0745
Center Mast	150	132-144 hr	1	7/7/2019	1818	7/8/2019	0743
Center Mast	150	132-144 hr	2	7/7/2019	1819	7/8/2019	0744
Center Mast	15	144-156 hr	1	7/8/2019	0756	7/8/2019	1802
Center Mast	33	144-156 hr	1	7/8/2019	0754	7/8/2019	1801
Center Mast	55	144-156 hr	1	7/8/2019	0752	7/8/2019	1800
Center Mast	90	144-156 hr	1	7/8/2019	0752	7/8/2019	1759
Center Mast	150	144-156 hr	1	7/8/2019	0750	7/8/2019	1757
Center Mast	150	144-156 hr	2	7/8/2019	0751	7/8/2019	1758
Center Mast	15	156-168 hr	1	7/8/2019	1812	7/9/2019	0739
Center Mast	33	156-168 hr	1	7/8/2019	1810	7/9/2019	0737
Center Mast	55	156-168 hr	1	7/8/2019	1809	7/9/2019	0737
Center Mast	90	156-168 hr	1	7/8/2019	1808	7/9/2019	0736
Center Mast	150	156-168 hr	1	7/8/2019	1806	7/9/2019	0734
Center Mast	150	156-168 hr	2	7/8/2019	1806	7/9/2019	0735
Perimeter Off-Field	A	0-6 hr	1	7/2/2019	1003	7/2/2019	1520
Perimeter Off-Field	B	0-6 hr	1	7/2/2019	1007	7/2/2019	1528
Perimeter Off-Field	C	0-6 hr	1	7/2/2019	1003	7/2/2019	1527
Perimeter Off-Field	D	0-6 hr	1	7/2/2019	1015	7/2/2019	1515
Perimeter Off-Field	E	0-6 hr	1	7/2/2019	1002	7/2/2019	1516
Perimeter Off-Field	F	0-6 hr	1	7/2/2019	1016	7/2/2019	1525
Perimeter Off-Field	G	0-6 hr	1	7/2/2019	1016	7/2/2019	1514
Perimeter Off-Field	H	0-6 hr	1	7/2/2019	1008	7/2/2019	1526
Perimeter Off-Field	A	6-12 hr	1	7/2/2019	1522	7/2/2019	1755
Perimeter Off-Field	B	6-12 hr	1	7/2/2019	1532	7/2/2019	1804
Perimeter Off-Field	C	6-12 hr	1	7/2/2019	1530	7/2/2019	1817

**Appendix E. Sample Period Collection Dates and Times (continued)**

Sample Description	Height (cm) or Location	Sample Period	Rep	Sample Start Date	Sample Start Time	Sample End Date	Sample End Time
Perimeter Off-Field	D	6-12 hr	1	7/2/2019	1519	7/2/2019	1807
Perimeter Off-Field	E	6-12 hr	1	7/2/2019	1520	7/2/2019	1755
Perimeter Off-Field	F	6-12 hr	1	7/2/2019	1529	7/2/2019	1803
Perimeter Off-Field	G	6-12 hr	1	7/2/2019	1516	7/2/2019	1757
Perimeter Off-Field	H	6-12 hr	1	7/2/2019	1527	7/2/2019	1806
Perimeter Off-Field	A	12-24 hr	1	7/2/2019	1759	7/3/2019	0745
Perimeter Off-Field	B	12-24 hr	1	7/2/2019	1807	7/3/2019	0752
Perimeter Off-Field	C	12-24 hr	1	7/2/2019	1824	7/3/2019	0745
Perimeter Off-Field	D	12-24 hr	1	7/2/2019	1811	7/3/2019	0756
Perimeter Off-Field	E	12-24 hr	1	7/2/2019	1758	7/3/2019	0746
Perimeter Off-Field	F	12-24 hr	1	7/2/2019	1806	7/3/2019	0751
Perimeter Off-Field	G	12-24 hr	1	7/2/2019	1757	7/3/2019	0747
Perimeter Off-Field	H	12-24 hr	1	7/2/2019	1808	7/3/2019	0756
Perimeter Off-Field	A	24-36 hr	1	7/3/2019	0748	7/3/2019	1637
Perimeter Off-Field	B	24-36 hr	1	7/3/2019	0756	7/3/2019	1645
Perimeter Off-Field	C	24-36 hr	1	7/3/2019	0750	7/3/2019	1639
Perimeter Off-Field	D	24-36 hr	1	7/3/2019	0758	7/3/2019	1649
Perimeter Off-Field	E	24-36 hr	1	7/3/2019	0748	7/3/2019	1637
Perimeter Off-Field	F	24-36 hr	1	7/3/2019	0754	7/3/2019	1649
Perimeter Off-Field	G	24-36 hr	1	7/3/2019	0748	7/3/2019	1639
Perimeter Off-Field	H	24-36 hr	1	7/3/2019	0757	7/3/2019	1653
Perimeter Off-Field	A	36-48 hr	1	7/3/2019	1640	7/4/2019	0733
Perimeter Off-Field	B	36-48 hr	1	7/3/2019	1649	7/4/2019	0742
Perimeter Off-Field	C	36-48 hr	1	7/3/2019	1643	7/4/2019	0738
Perimeter Off-Field	D	36-48 hr	1	7/3/2019	1652	7/4/2019	0740
Perimeter Off-Field	E	36-48 hr	1	7/3/2019	1646	7/4/2019	0737
Perimeter Off-Field	F	36-48 hr	1	7/3/2019	1652	7/4/2019	0743
Perimeter Off-Field	G	36-48 hr	1	7/3/2019	1642	7/4/2019	0734
Perimeter Off-Field	H	36-48 hr	1	7/3/2019	1655	7/4/2019	0742
Perimeter Off-Field	A	48-60 hr	1	7/4/2019	0735	7/4/2019	1724
Perimeter Off-Field	B	48-60 hr	1	7/4/2019	0744	7/4/2019	1730
Perimeter Off-Field	C	48-60 hr	1	7/4/2019	0740	7/4/2019	1726
Perimeter Off-Field	D	48-60 hr	1	7/4/2019	0750	7/4/2019	1734
Perimeter Off-Field	E	48-60 hr	1	7/4/2019	0739	7/4/2019	1727
Perimeter Off-Field	F	48-60 hr	1	7/4/2019	0745	7/4/2019	1733

**Appendix E. Sample Period Collection Dates and Times (continued)**

Sample Description	Height (cm) or Location	Sample Period	Rep	Sample Start Date	Sample Start Time	Sample End Date	Sample End Time
Perimeter Off-Field	G	48-60 hr	1	7/4/2019	0735	7/4/2019	1728
Perimeter Off-Field	H	48-60 hr	1	7/4/2019	0743	7/4/2019	1737
Perimeter Off-Field	A	60-72 hr	1	7/4/2019	1726	7/5/2019	0739
Perimeter Off-Field	B	60-72 hr	1	7/4/2019	1733	7/5/2019	0745
Perimeter Off-Field	C	60-72 hr	1	7/4/2019	1729	7/5/2019	0739
Perimeter Off-Field	D	60-72 hr	1	7/4/2019	1739	7/5/2019	0747
Perimeter Off-Field	E	60-72 hr	1	7/4/2019	1729	7/5/2019	0741
Perimeter Off-Field	F	60-72 hr	1	7/4/2019	1736	7/5/2019	0747
Perimeter Off-Field	G	60-72 hr	1	7/4/2019	1730	7/5/2019	0741
Perimeter Off-Field	H	60-72 hr	1	7/4/2019	1739	7/5/2019	0748
Perimeter Off-Field	A	72-84 hr	1	7/5/2019	0741	7/5/2019	1627
Perimeter Off-Field	B	72-84 hr	1	7/5/2019	0747	7/5/2019	1632
Perimeter Off-Field	C	72-84 hr	1	7/5/2019	0742	7/5/2019	1628
Perimeter Off-Field	D	72-84 hr	1	7/5/2019	0749	7/5/2019	1633
Perimeter Off-Field	E	72-84 hr	1	7/5/2019	0742	7/5/2019	1627
Perimeter Off-Field	F	72-84 hr	1	7/5/2019	0749	7/5/2019	1635
Perimeter Off-Field	G	72-84 hr	1	7/5/2019	0743	7/5/2019	1626
Perimeter Off-Field	H	72-84 hr	1	7/5/2019	0749	7/5/2019	1634
Perimeter Off-Field	A	84-96 hr	1	7/5/2019	1629	7/6/2019	0758
Perimeter Off-Field	B	84-96 hr	1	7/5/2019	1635	7/6/2019	0803
Perimeter Off-Field	C	84-96 hr	1	7/5/2019	1630	7/6/2019	0757
Perimeter Off-Field	D	84-96 hr	1	7/5/2019	1636	7/6/2019	0804
Perimeter Off-Field	E	84-96 hr	1	7/5/2019	1631	7/6/2019	0756
Perimeter Off-Field	F	84-96 hr	1	7/5/2019	1637	7/6/2019	0802
Perimeter Off-Field	G	84-96 hr	1	7/5/2019	1628	7/6/2019	0756
Perimeter Off-Field	H	84-96 hr	1	7/5/2019	1636	7/6/2019	0810
Perimeter Off-Field	A	96-108 hr	1	7/6/2019	0800	7/6/2019	1801
Perimeter Off-Field	B	96-108 hr	1	7/6/2019	0805	7/6/2019	1807
Perimeter Off-Field	C	96-108 hr	1	7/6/2019	0759	7/6/2019	1801
Perimeter Off-Field	D	96-108 hr	1	7/6/2019	0805	7/6/2019	1810
Perimeter Off-Field	E	96-108 hr	1	7/6/2019	0758	7/6/2019	1802
Perimeter Off-Field	F	96-108 hr	1	7/6/2019	0804	7/6/2019	1808
Perimeter Off-Field	G	96-108 hr	1	7/6/2019	0758	7/6/2019	1803
Perimeter Off-Field	H	96-108 hr	1	7/6/2019	0811	7/6/2019	1811



**Appendix E. Sample Period Collection Dates and Times (continued)**

Sample Description	Height (cm) or Location	Sample Period	Rep	Sample Start Date	Sample Start Time	Sample End Date	Sample End Time
Perimeter Off-Field	A	108-120 hr	1	7/6/2019	1803	7/7/2019	0737
Perimeter Off-Field	B	108-120 hr	1	7/6/2019	1809	7/7/2019	0741
Perimeter Off-Field	C	108-120 hr	1	7/6/2019	1805	7/7/2019	0736
Perimeter Off-Field	D	108-120 hr	1	7/6/2019	1812	7/7/2019	0744
Perimeter Off-Field	E	108-120 hr	1	7/6/2019	1804	7/7/2019	0736
Perimeter Off-Field	F	108-120 hr	1	7/6/2019	1810	7/7/2019	0742
Perimeter Off-Field	G	108-120 hr	1	7/6/2019	1805	7/7/2019	0740
Perimeter Off-Field	H	108-120 hr	1	7/6/2019	1813	7/7/2019	0747
Perimeter Off-Field	A	120-132 hr	1	7/7/2019	0739	7/7/2019	1803
Perimeter Off-Field	B	120-132 hr	1	7/7/2019	0743	7/7/2019	1809
Perimeter Off-Field	C	120-132 hr	1	7/7/2019	0738	7/7/2019	1806
Perimeter Off-Field	D	120-132 hr	1	7/7/2019	0746	7/7/2019	1815
Perimeter Off-Field	E	120-132 hr	1	7/7/2019	0738	7/7/2019	1805
Perimeter Off-Field	F	120-132 hr	1	7/7/2019	0743	7/7/2019	1812
Perimeter Off-Field	G	120-132 hr	1	7/7/2019	0742	7/7/2019	1805
Perimeter Off-Field	H	120-132 hr	1	7/7/2019	0748	7/7/2019	1813
Perimeter Off-Field	A	132-144 hr	1	7/7/2019	1806	7/8/2019	0741
Perimeter Off-Field	B	132-144 hr	1	7/7/2019	1815	7/8/2019	0746
Perimeter Off-Field	C	132-144 hr	1	7/7/2019	1809	7/8/2019	0743
Perimeter Off-Field	D	132-144 hr	1	7/7/2019	1820	7/8/2019	0750
Perimeter Off-Field	E	132-144 hr	1	7/7/2019	1808	7/8/2019	0741
Perimeter Off-Field	F	132-144 hr	1	7/7/2019	1814	7/8/2019	0747
Perimeter Off-Field	G	132-144 hr	1	7/7/2019	1807	7/8/2019	0741
Perimeter Off-Field	H	132-144 hr	1	7/7/2019	1815	7/8/2019	0746
Perimeter Off-Field	A	144-156 hr	1	7/8/2019	0742	7/8/2019	1756
Perimeter Off-Field	B	144-156 hr	1	7/8/2019	0747	7/8/2019	1801
Perimeter Off-Field	C	144-156 hr	1	7/8/2019	0744	7/8/2019	1755
Perimeter Off-Field	D	144-156 hr	1	7/8/2019	0752	7/8/2019	1804
Perimeter Off-Field	E	144-156 hr	1	7/8/2019	0743	7/8/2019	1756
Perimeter Off-Field	F	144-156 hr	1	7/8/2019	0748	7/8/2019	1802
Perimeter Off-Field	G	144-156 hr	1	7/8/2019	0742	7/8/2019	1757
Perimeter Off-Field	H	144-156 hr	1	7/8/2019	0747	7/8/2019	1803
Perimeter Off-Field	A	156-168 hr	1	7/8/2019	1758	7/9/2019	0732
Perimeter Off-Field	B	156-168 hr	1	7/8/2019	1803	7/9/2019	0736
Perimeter Off-Field	C	156-168 hr	1	7/8/2019	1759	7/9/2019	0734

**Appendix E. Sample Period Collection Dates and Times (continued)**

<b>Sample Description</b>	<b>Height (cm) or Location</b>	<b>Sample Period</b>	<b>Rep</b>	<b>Sample Start Date</b>	<b>Sample Start Time</b>	<b>Sample End Date</b>	<b>Sample End Time</b>
Perimeter Off-Field	D	156-168 hr	1	7/8/2019	1807	7/9/2019	0737
Perimeter Off-Field	E	156-168 hr	1	7/8/2019	1758	7/9/2019	0733
Perimeter Off-Field	F	156-168 hr	1	7/8/2019	1804	7/9/2019	0737
Perimeter Off-Field	G	156-168 hr	1	7/8/2019	1759	7/9/2019	0732
Perimeter Off-Field	H	156-168 hr	1	7/8/2019	1805	7/9/2019	0737
Field QC Spikes	0 ng	6 hr	1	7/7/2019	1540	7/7/2019	2143
Field QC Spikes	0 ng	6 hr	2	7/7/2019	1540	7/7/2019	2143
Field QC Spikes	0 ng	6 hr	3	7/7/2019	1540	7/7/2019	2143
Field QC Spikes	3 ng	6 hr	1	7/7/2019	1543	7/7/2019	2145
Field QC Spikes	3 ng	6 hr	2	7/7/2019	1543	7/7/2019	2145
Field QC Spikes	3 ng	6 hr	3	7/7/2019	1543	7/7/2019	2145
Field QC Spikes	10 ng	6 hr	1	7/7/2019	1544	7/7/2019	2146
Field QC Spikes	10 ng	6 hr	2	7/7/2019	1544	7/7/2019	2146
Field QC Spikes	10 ng	6 hr	3	7/7/2019	1544	7/7/2019	2146
Field QC Spikes	30 ng	6 hr	1	7/7/2019	1545	7/7/2019	2148
Field QC Spikes	30 ng	6 hr	2	7/7/2019	1545	7/7/2019	2148
Field QC Spikes	30 ng	6 hr	3	7/7/2019	1545	7/7/2019	2148
Field QC Spikes	0 ng	12 hr	1	7/6/2019	1641	7/7/2019	0502
Field QC Spikes	0 ng	12 hr	2	7/6/2019	1641	7/7/2019	0502
Field QC Spikes	0 ng	12 hr	3	7/6/2019	1641	7/7/2019	0502
Field QC Spikes	3 ng	12 hr	1	7/6/2019	1645	7/7/2019	0505
Field QC Spikes	3 ng	12 hr	2	7/6/2019	1645	7/7/2019	0505
Field QC Spikes	3 ng	12 hr	3	7/6/2019	1645	7/7/2019	0505
Field QC Spikes	10 ng	12 hr	1	7/6/2019	1647	7/7/2019	0506
Field QC Spikes	10 ng	12 hr	2	7/6/2019	1647	7/7/2019	0506
Field QC Spikes	10 ng	12 hr	3	7/6/2019	1647	7/7/2019	0506
Field QC Spikes	30 ng	12 hr	1	7/6/2019	1648	7/7/2019	0507
Field QC Spikes	30 ng	12 hr	2	7/6/2019	1648	7/7/2019	0507
Field QC Spikes	30 ng	12 hr	3	7/6/2019	1648	7/7/2019	0507

**Appendix E. Sample Period Collection Dates and Times (continued)**

Sample Description	Event	Transect	Sample Start Date	Placement Start Time	Placement End Time	Sample End Date	Removal Start Time	Removal End Time
Filter Paper	1HR	UWA	7/2/2019	- <sup>1</sup>	- <sup>1</sup>	7/2/2019	1030	1033
Filter Paper	1HR	UWB	7/2/2019	-	-	7/2/2019	1023	1027
Filter Paper	1HR	RWA	7/2/2019	-	-	7/2/2019	1044	1048
Filter Paper	1HR	RWB	7/2/2019	-	-	7/2/2019	1027	1034
Filter Paper	1HR	DWA	7/2/2019	-	-	7/2/2019	1011	1015
Filter Paper	1HR	DWB	7/2/2019	-	-	7/2/2019	1022	1026
Filter Paper	1HR	DWC	7/2/2019	-	-	7/2/2019	1028	1032
Filter Paper	1HR	LWA	7/2/2019	-	-	7/2/2019	1033	1038
Filter Paper	1HR	LWB	7/2/2019	-	-	7/2/2019	1023	1029
Filter Paper	24HR	UWA	7/2/2019	1104	1111	7/3/2019	0825	0831
Filter Paper	24HR	UWB	7/2/2019	1056	1101	7/3/2019	0812	0820
Filter Paper	24HR	RWA	7/2/2019	1051	1054	7/3/2019	0851	0857
Filter Paper	24HR	RWB	7/2/2019	1056	1059	7/3/2019	0839	0847
Filter Paper	24HR	DWA	7/2/2019	1051	1055	7/3/2019	0807	0815
Filter Paper	24HR	DWB	7/2/2019	1057	1101	7/3/2019	0817	0825
Filter Paper	24HR	DWC	7/2/2019	1103	1107	7/3/2019	0827	0835
Filter Paper	24HR	LWA	7/2/2019	1106	1109	7/3/2019	0838	0848
Filter Paper	24HR	LWB	7/2/2019	1059	1102	7/3/2019	0857	0904
Filter Paper	48HR	UWA	7/3/2019	0927	0930	7/4/2019	0813	0820
Filter Paper	48HR	UWB	7/3/2019	0937	0941	7/4/2019	0802	0808
Filter Paper	48HR	RWA	7/3/2019	0905	0908	7/4/2019	0846	0854
Filter Paper	48HR	RWB	7/3/2019	0913	0916	7/4/2019	0836	0842
Filter Paper	48HR	DWA	7/3/2019	0927	0930	7/4/2019	0755	0804
Filter Paper	48HR	DWB	7/3/2019	0923	0926	7/4/2019	0807	0814
Filter Paper	48HR	DWC	7/3/2019	0918	0922	7/4/2019	0819	0829
Filter Paper	48HR	LWA	7/3/2019	0921	0924	7/4/2019	0827	0835
Filter Paper	48HR	LWB	7/3/2019	0915	0918	7/4/2019	0850	0857
Filter Paper	72HR	UWA	7/4/2019	0923	0926	7/5/2019	0811	0818
Filter Paper	72HR	UWB	7/4/2019	0929	0933	7/5/2019	0802	0807
Filter Paper	72HR	RWA	7/4/2019	0904	0906	7/5/2019	0834	0838
Filter Paper	72HR	RWB	7/4/2019	0909	0911	7/5/2019	0826	0830
Filter Paper	72HR	DWA	7/4/2019	0923	0926	7/5/2019	0758	0805
Filter Paper	72HR	DWB	7/4/2019	0919	0922	7/5/2019	0807	0813
Filter Paper	72HR	DWC	7/4/2019	0914	0917	7/5/2019	0816	0822
Filter Paper	72HR	LWA	7/4/2019	0914	0919	7/5/2019	0824	0828
Filter Paper	72HR	LWB	7/4/2019	0905	0909	7/5/2019	0834	0839

**Appendix E. Sample Period Collection Dates and Times (continued)**

<b>Sample Description</b>	<b>Event</b>	<b>Transect</b>	<b>Sample Start Date</b>	<b>Placement Start Time</b>	<b>Placement End Time</b>	<b>Sample End Date</b>	<b>Removal Start Time</b>	<b>Removal End Time</b>
Filter Paper	96HR	UWA	7/5/2019	0901	0903	7/6/2019	0831	0840
Filter Paper	96HR	UWB	7/5/2019	0906	0908	7/6/2019	0819	0827
Filter Paper	96HR	RWA	7/5/2019	0846	0848	7/6/2019	0902	0907
Filter Paper	96HR	RWB	7/5/2019	0850	0852	7/6/2019	0854	0859
Filter Paper	96HR	DWA	7/5/2019	0902	0905	7/6/2019	0820	0826
Filter Paper	96HR	DWB	7/5/2019	0858	0901	7/6/2019	0830	0835
Filter Paper	96HR	DWC	7/5/2019	0855	0857	7/6/2019	0839	0845
Filter Paper	96HR	LWA	7/5/2019	0854	0856	7/6/2019	0846	0851
Filter Paper	96HR	LWB	7/5/2019	0848	0851	7/6/2019	0859	0903
Filter Paper	120HR	UWA	7/6/2019	0927	0930	7/7/2019	0807	0811
Filter Paper	120HR	UWB	7/6/2019	0934	0938	7/7/2019	0757	0801
Filter Paper	120HR	RWA	7/6/2019	0914	0916	7/7/2019	0825	0829
Filter Paper	120HR	RWB	7/6/2019	0918	0920	7/7/2019	0820	0823
Filter Paper	120HR	DWA	7/6/2019	0932	0935	7/7/2019	0756	0800
Filter Paper	120HR	DWB	7/6/2019	0927	0931	7/7/2019	0803	0807
Filter Paper	120HR	DWC	7/6/2019	0923	0926	7/7/2019	0810	0814
Filter Paper	120HR	LWA	7/6/2019	0918	0921	7/7/2019	0816	0821
Filter Paper	120HR	LWB	7/6/2019	0909	0913	7/7/2019	0825	0831
Filter Paper	144HR	UWA	7/7/2019	0848	0851	7/8/2019	0815	0820
Filter Paper	144HR	UWB	7/7/2019	0855	0858	7/8/2019	0806	0811
Filter Paper	144HR	RWA	7/7/2019	0837	0839	7/8/2019	0827	0831
Filter Paper	144HR	RWB	7/7/2019	0841	0843	7/8/2019	0819	0823
Filter Paper	144HR	DWA	7/7/2019	0855	0857	7/8/2019	0754	0759
Filter Paper	144HR	DWB	7/7/2019	0851	0853	7/8/2019	0802	0807
Filter Paper	144HR	DWC	7/7/2019	0846	0849	7/8/2019	0810	0813
Filter Paper	144HR	LWA	7/7/2019	0841	0844	7/8/2019	0825	0830
Filter Paper	144HR	LWB	7/7/2019	0835	0838	7/8/2019	0835	0840
Filter Paper	168HR	UWA	7/8/2019	0856	0858	7/9/2019	0758	0803
Filter Paper	168HR	UWB	7/8/2019	0901	0903	7/9/2019	0748	0753
Filter Paper	168HR	RWA	7/8/2019	0837	0838	7/9/2019	0816	0822
Filter Paper	168HR	RWB	7/8/2019	0840	0842	7/9/2019	0810	0814
Filter Paper	168HR	DWA	7/8/2019	0852	0854	7/9/2019	0744	0749
Filter Paper	168HR	DWB	7/8/2019	0848	0850	7/9/2019	0751	0757
Filter Paper	168HR	DWC	7/8/2019	0844	0847	7/9/2019	0801	0806
Filter Paper	168HR	LWA	7/8/2019	0850	0853	7/9/2019	0808	0813
Filter Paper	168HR	LWB	7/8/2019	0845	0848	7/9/2019	0818	0824

<sup>1</sup> One hour filter papers were placed in the field prior to application.

## **Appendix C. Eurofins EAG Agroscience, LLC Analytical Sub-Report**

## **ANALYTICAL SUB-REPORT**

### **TITLE**

Off-target Movement Assessment of a Spray Solution Containing  
MON 76980 + MON 79789 + Intact™ + MON 51817 - Illinois

### **TEST GUIDELINES**

US EPA OPPTS 835.8100: Field Volatility  
US EPA OPPTS 840.1200: Spray Drift Field Deposition

### **AUTHOR**

Matthew Rebstock

### **ANALYTICAL SUB-REPORT COMPLETION DATE**

December 23, 2019

### **SPONSOR**

Monsanto Company  
700 Chesterfield Parkway West  
Chesterfield, MO 63017

### **ANALYTICAL PERFORMING LABORATORY**

Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202

### **STUDY DIRECTOR/TESTING FACILITY**

Will Griesse  
Monsanto Company  
700 Chesterfield Parkway West  
Chesterfield, MO 63017

### **SPONSOR STUDY NUMBER**

REG-2019-0035

### **EUROFINS STUDY NUMBER**


89311

### **TOTAL PAGE COUNT**

Page 1 of 162

### COMPLIANCE STATEMENT

The analytical phase of this study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency 40 CFR Part 160 (1989); which are compatible with the Organisation for Economic Cooperation and Development (OECD) Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17).



---

Matthew Rebstock  
Analytical Chemistry Principal Investigator  
Eurofins EAG Agrosience, LLC

23 DEC 2019  
Date

## QUALITY ASSURANCE STATEMENT

**STUDY TITLE:** Off-target Movement Assessment of a Spray Solution  
Containing MON 76980 + MON 79789 Intact™ +  
MON 51817 - Illinois

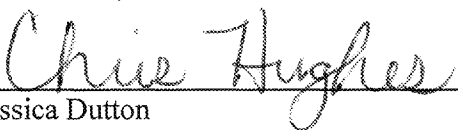
**Study Number:** REG-2019-0035

**Eurofins Study Number:** 89311

Audit inspection reports have been submitted to the Study Director and Management documenting the status of compliance with applicable departmental standard operating procedures, the study protocol, and Good Laboratory Practice regulations. The inspections conducted by Eurofins Quality Assurance Unit are listed below:

Dates of Inspection/Audit	Phase	Date Reported to Study Director	Date Reported to SD Management
18 Jul 2019	Procedure: Sample Filtering and Aliquoting	26 Aug 2019	26 Aug 2019
18 Jul 2019	Procedure: Dose Solution Preparation	26 Aug 2019	26 Aug 2019
19 Jul 2019	Procedure: Tank Mix QC Weigh Outs	26 Aug 2019	26 Aug 2019
23-26, 28, 29 Jul 2019	Raw Data	10 Oct 2019	10 Oct 2019
24-25 Oct 2019	Raw Data and Draft Report	23 Dec 2019	23 Dec 2019
23 Dec 2019	Final Analytical Report	23 Dec 2019	23 Dec 2019

Reviews conducted by Eurofins Quality Assurance confirm that this sub-report accurately describes the methods and standard operating procedures followed and accurately reflect the raw data for the portion of the study conducted by Eurofins.


 FOR 23 Dec 2019  
\_\_\_\_\_  
Jessica Dutton Date  
Manager, Quality Assurance  
Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202



### CERTIFICATION PAGE

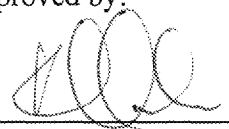
This report is an accurate and complete representation of the Eurofins EAG Agrosience, LLC analytical phase of study REG-2019-0035.

Analytical Chemistry Principal Investigator/Author:

  
\_\_\_\_\_  
Matthew Rebstock  
Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202  
Phone: 573-777-6385  
E-mail: matthewrebstock@eurofinsUS.com

23 Dec 2019  
\_\_\_\_\_  
Date

Approved by:

  
\_\_\_\_\_  
Kevin Clark  
Principal Director/Business Unit Leader  
Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202  
Phone: 573-777-6379  
E-mail: kevinclark@eurofinsUS.com

23 Dec 2019  
\_\_\_\_\_  
Date

## STUDY INFORMATION

Analytical Sub-Report Title: Off-target Movement Assessment of a Spray Solution Containing MON 76980 + MON 79789 + Intact™ + MON 51817 - Illinois

Study Number: REG-2019-0035

Eurofins Study Number: 89311

Study Director: Will Griese

Analytical Chemistry  
Principal Investigator  
and Author: Matthew Rebstock

Analytical Phase  
Performing Laboratory: Eurofins EAG Agrosience, LLC  
7200 East ABC Lane  
Columbia, MO 65202

Study Personnel: Bryanne Cornine, Staff Scientist I  
Wesley Fain, Senior Scientist/Team Lead  
Courtney Travis, Staff Scientist I

Sample Analysis  
Start Date: July 15, 2019

Sample Analysis  
Termination Date: July 20, 2019

Analytical Report  
Completion Date: December 23, 2019

Archiving of Records: The original Analytical Sub-Report, analytical raw data, and records pertaining to this phase of the study will be retained in the Eurofins archives in Columbia, MO until transferred to the Monsanto Regulatory Archives in St. Louis, MO as appropriate. Copies of the original analytical sub-report and paper raw data as well as the electronic raw data pertaining to this phase of the study will be retained in the Eurofins archives in Columbia, MO. All samples not consumed upon analysis will be discarded after acceptance of the report by the Sponsor and Q.A. verification for GLP compliance and approval to discard has been given by the study director.

© 2019 Bayer Group. All Rights Reserved.

*This document is protected under national and international copyright law and treaties. This document and any accompanying material are for use only by the regulatory authority to which it has been submitted by Monsanto Company and its affiliates, collectively "Bayer Group", and only in support of actions requested by Bayer Group. Any other use, copying, or transmission, including internet posting, of this document and the materials described in or accompanying this document, without prior consent of Monsanto Company, is strictly prohibited; except that Monsanto Company hereby grants such consent to the regulatory authority where required under applicable law or regulation. The intellectual property, information and materials described in or accompanying this document are owned by Bayer Group, which has filed for or been granted patents on those materials. By submitting this document and any accompanying materials, Monsanto Company and the Bayer Group do not grant any party or entity any right or license to the information, material or intellectual property described or contained in this submission.*

## ABBREVIATIONS AND ACRONYMS

a.e.:	acid equivalent
Amt:	amount
Aliq:	aliquot
CFR:	Code of Federal Regulations
Conc:	concentration
Corr:	corrected
EEAL:	Eurofins EAG Agrosience, LLC
EPA:	Environmental Protection Agency
FIFRA:	Federal Insecticide, Fungicide, and Rodenticide Act
Fort:	fortification
g:	gram
GLP:	good laboratory practices
HPLC:	High Performance Liquid Chromatography
ID:	identification
LC-MS/MS:	liquid chromatography tandem mass spectrometry
LOD:	limit of detection
LOQ:	limit of quantitation
µg	microgram
mg:	milligram
mm:	millimeter
ng:	nanogram
nm:	nanometer
No:	number
Nom:	nominal
PUF:	polyurethane foam
RSD:	relative standard deviation
Rec:	recovery
Sol:	solution
Std. Dev:	standard deviation
Trt.	treatment
U.S.:	United States
UV:	ultraviolet
wt:	weight

## TABLE OF CONTENTS

Title Page .....	1
Compliance Statement .....	2
Quality Assurance Statement .....	3
Certification Page .....	4
Study Information .....	5
Abbreviations and Acronyms .....	7
Table of Contents .....	8
1. Summary .....	10
2. Purpose .....	10
3. Materials .....	10
3.1 Reference Substance .....	10
3.2 Internal Standard .....	11
3.3 Surrogate Formulation .....	11
3.4 Stability of Reference Standard and Solutions .....	11
3.5 Formulations Used During the Study .....	12
4. Analytical Methods .....	12
4.1 Analysis of Application Monitoring Filter Papers for Dicamba .....	12
4.2 Analysis of Tank Mix Samples for Dicamba .....	12
4.3 Determination of Dicamba on PUFs .....	13
4.3.1 Analytical Method Summary .....	13
4.3.2 On-going Method Performance Verification .....	13
4.3.3 PUF Sample Preparation and Storage .....	13
4.3.4 PUF Sample Analysis .....	13
5. Analytical Deviations .....	13
6. Results .....	14
6.1 Application Monitoring Filter Paper Sample Results .....	14
6.2 Tank Mix Sample Results .....	14
6.3 PUF Sample Results .....	14
6.3.1 Procedural Recoveries .....	14
6.3.2 Individual PUF Sample Results .....	15
6.3.3 Field Exposed and Transit Stability PUF Sample Results .....	15
7. References .....	16
8. Tables .....	17
Table 1. Recovery of Dicamba from Fortified Application Monitoring Filter Paper Samples .....	18
Table 2. Application Monitoring Filter Paper Sample Results .....	19
Table 3. Recovery of Dicamba from Fortified Tank Mix Samples .....	20
Table 4. Tank Mix Sample Results .....	21
Table 5. Recovery of Dicamba from Fortified Polyurethane Foam Samples .....	22
Table 6. Center Mast PUF Sample Results .....	23
Table 7. Perimeter PUF Sample Results .....	26
Table 8. Field Exposed PUF Sample Results .....	30
Table 9. Transit Stability PUF Sample Results .....	31
9. Figures .....	32
Figure 1. Chemical Structure of Reference Substance .....	32

Figure 2.	Chemical Structure of Internal Standard .....	32
10.	Appendices.....	33
Appendix A.	Certificates of Analysis for the Reference Substance and Internal Standard.....	33
Appendix B.	Analytical Method – Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification .....	36
Appendix C.	Analytical Method – Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification .....	45
Appendix D.	Analytical Method – LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps.....	53
Appendix E.	Raw Data, Recoveries, and Statistics for Application Monitoring Filter Paper Analysis.....	74
E.1	Recovery Data Table and Statistics for Application Monitoring Filter Paper Samples Fortified with Dicamba.....	75
E.2	Raw Data Table for Application Monitoring Filter Paper Sample Analysis.....	79
Appendix F.	Raw Data, Recoveries, and Statistics for Tank Mix Sample Analysis.....	82
F.1	Recovery Data and Statistics for Tank Mix Samples Fortified with Dicamba .....	83
F.2	Raw Data Table for Tank Mix Sample Analysis.....	87
Appendix G.	Raw Data, Recoveries, and Statistics for PUF Sample Analysis .....	90
G.1	Recovery Data Table and Statistics for PUF Samples Fortified with Dicamba .....	91
G.2	Raw Data Table for PUF Sample Analysis.....	97
Appendix H.	Significant Dates Table .....	109
Appendix I.	Representative Chromatograms and Calibration Curve – Dicamba on Application Monitoring Filter Paper and Tank Mix .....	117
Appendix J.	Representative Chromatograms – Dicamba on Application Monitoring Filter Paper .....	122
Appendix K.	Representative Chromatograms – Dicamba in Tank Mix .....	125
Appendix L.	Representative Chromatograms and Calibration Curve – Dicamba on PUF.....	127
L.1	Representative Chromatograms and Calibration Curve – Dicamba on PUF .....	128
L.2	Representative Chromatograms of Concurrent Recoveries of Dicamba from Untreated and Fortified PUF Samples.....	134
L.3	Representative Chromatograms of Dicamba PUF Samples .....	140
L.4	Representative Chromatograms of Field Exposed Spike and Transit Stability PUF Samples.....	149
Appendix M.	Analysis Conditions .....	160

## 1. SUMMARY

The work reported here is the analytical phase of study REG-2019-0035 performed by Eurofins EAG Agrosience, LLC. The purpose of the analytical phase of this study was to quantitate the primary active ingredient dicamba in different media after application of a tank mix containing the test substance MON 76980 and additional tank mix partners.

The test system used in this study was a test plot planted with both dicamba tolerant and non-dicamba tolerant soybeans. The PUF collectors (22 mm diameter × 76 mm length) were placed at specific heights/distances within and around the field and used to assess the field volatility of dicamba. Four (4) sets of four (4) Whatman #3 filter papers (12.5 cm diameter) were affixed to horizontal stands within the spray swath to verify application rates. Tank mix samples were also used to verify application rates.

Dicamba on application monitoring filter paper samples was analyzed using the current version of analytical method ME-2166. Method performance assessments within the study showed acceptable accuracy (average within 90-110%) and precision ( $\leq 5\%$  RSD) for application monitoring filter paper analyses.

Dicamba in tank mix samples was analyzed using the current version of analytical method ME-2154 with Eurofins modifications. Method performance assessments within the study showed acceptable accuracy (average within 90-110%) and precision ( $\leq 5\%$  RSD) for tank mix analyses.

Dicamba on PUF collectors was analyzed using the current version of analytical method ME-2242. In this study, the working range of the analytical method was 0.030 to 7.5 ng/PUF with a Limit of Quantitation (LOQ) of 0.1 ng/PUF. Method performance assessments within the study showed acceptable accuracy (average within 70-120%), precision ( $\leq 20\%$  RSD), and selectivity ( $\leq 30\%$  of the LOQ) for PUF collectors fortified with dicamba, with the exception of analytical Set 001 in which the LOQ fortifications resulted in an RSD of 20.7%.

## 2. PURPOSE

The purpose of this analytical phase of this study was to measure dicamba volatilization (mass of dicamba per PUF collector) following the application of a tank mix containing dicamba formulation MON 76980. Analytical work was conducted under the U.S. EPA FIFRA Good Laboratory Practice Standards.

## 3. MATERIALS

### 3.1 Reference Substance

The analytical reference standard used in this study was as follows:

Common Name:	Dicamba
Chemical Name:	3,6-dichloro-2-methoxybenzoic acid
CAS Registry Number:	1918-00-9
Molecular Weight:	221.04 g/mol
Purity:	99.4%
Lot Number:	109334

The amount of reference substance weighed out for each stock solution was corrected for the purity of the reference substance. The chemical structure of the reference substance is provided in [Figure 1](#).

### 3.2 Internal Standard

Common Name:	$^{13}\text{C}_6$ -Dicamba
Chemical Name:	3,6-dichloro-2-methoxybenzoic-1,2,3,4,5,6- $^{13}\text{C}_6$ acid
CAS Registry Number:	1173023-06-7
Molecular Weight:	227.04 g/mol
Lot Number:	109335

The chemical structure of the internal standard is provided in [Figure 2](#).

### 3.3 Surrogate Formulation

Formulation:	MON 76981
Active Ingredient 1:	diglycolamine salt of dicamba
Purity:	9.84% wt. (a.e.)
Active Ingredient 2:	monoethanolamine salt of glyphosate
Purity:	19.16% wt. (a.e.)
Lot Number:	11448162

The certificates of analysis for the reference substance, internal standard, and surrogate formulation are in [Appendix A](#).

### 3.4 Stability of Reference Standard and Solutions

The reference standard was obtained from the Monsanto Analytical Reference Standards Program and was stored under the conditions recommended on the corresponding certificates of analysis. Stability of the neat reference standard was monitored as part of the Analytical Reference Standards Program and was certified per the SOP for the program (PR-0522).

In this study, three analytical methods were used. Filter paper samples used for application monitoring were analyzed as per ME-2166 ([Appendix B](#)), tank mix samples were analyzed as per ME-2154 ([Appendix C](#)) with modifications, and PUF samples were analyzed as per ME-2242 ([Appendix D](#)).

The standard solutions used during analysis of the application monitoring pads (filter papers) and tank mix have a recommended six (6) month shelf life when stored refrigerated. All analyses were completed within this six (6) month period. See the analytical methods in [Appendix B](#) and [Appendix C](#) for specifications for preparation, storage and use of the calibration and fortification solutions.

Stock, intermediate, and working solutions used during analysis of PUF samples were stored in a monitored refrigerator at approximately  $\leq 8^\circ\text{C}$  (average daily range of 2 to  $8^\circ\text{C}$ ). Solutions containing dicamba prepared in absolute ethanol or acetonitrile and stored at approximately  $4^\circ\text{C}$  have been demonstrated to be stable for at least 201 days. All analyses were completed within this 201-day period. See the analytical method in [Appendix D](#) for specifications for preparation, storage, and use of the calibration and fortification solutions.

Calibration curves utilized for PUF sample analysis were generated from calibration standard solutions corresponding to concentrations (on matrix) from 0.030 to 7.5 ng/PUF



of dicamba. All calibration standard solutions were used within the demonstrated period of stability for dicamba.

### 3.5 Formulations Used During the Study

Formulation	MON 76980
Active Ingredient	Diglycolamine salt of dicamba
Nominal Loading (% a.e.)	29.0%

## 4. ANALYTICAL METHODS

### 4.1 Analysis of Application Monitoring Filter Papers for Dicamba

Horizontal platforms containing filter papers were placed within the field plot prior to application of dicamba formulation MON 76980, collected after spray and then analyzed to verify that the target dicamba concentration was sprayed. Four groups of four platforms with each platform holding one filter paper were used in the study. Each filter paper was an individual study sample for a total of 16 samples.

An HPLC analysis was conducted using the current version of method ME-2166 ([Appendix B](#)) to determine the amount of dicamba sprayed on each filter paper. Each filter paper sample was individually extracted with water. Standard curves for sample analyses were generated from standard solutions in concentrations from 5.26 to 253 µg/g dicamba. The surrogate formulation MON 76981 was used to prepare quality check samples targeting 75% (0.516 mg dicamba/filter) and 125% (0.860 mg dicamba/filter) of the expected concentration of test samples (0.688 mg dicamba/filter). Analysis conditions are presented in [Appendix M](#).

The statistical methods used in this study were calculations of the mean, standard deviation, and relative standard deviation. A summary of recoveries from laboratory prepared quality check filter paper samples is located in [Table 1](#). The recoveries of dicamba from each filter paper sample are summarized in [Table 2](#). Detailed recovery results can be found in [Appendix E](#).

### 4.2 Analysis of Tank Mix Samples for Dicamba

An HPLC analysis was conducted using method ME-2154 ([Appendix C](#)) with modifications to determine the amount of dicamba in tank mix samples. Samples were gently shaken to homogeneity and approximately 0.5 g of each sample were diluted with a sufficient amount of water to a final concentration of approximately 0.01% dicamba. Standard curves for sample analyses were generated from standard solutions in concentrations from 5.26 to 253 µg/g dicamba. The surrogate formulation MON 76981 was used to prepare quality check samples targeting 75% (0.30 wt% dicamba) and 125% (0.50 wt% dicamba) of the expected concentration of test samples (0.40 wt% dicamba). Analysis conditions are presented in [Appendix M](#).

The statistical methods used in this study were calculations of the mean of dicamba percent weight, standard deviation, and relative standard deviation. A summary of recoveries from laboratory prepared quality check tank mix samples is located in [Table 3](#). The concentrations of dicamba from six (6) tank mix samples are summarized in [Table 4](#). Detailed recovery results can be found in [Appendix F](#).

#### 4.3 Determination of Dicamba on PUFs

##### 4.3.1 Analytical Method Summary

Dicamba was extracted from the polyurethane foam (PUF) collectors using methanol in the presence of stable-labeled internal standard as per the current version of the analytical method ME-2242 ([Appendix D](#)). The sample tubes were agitated on a high-speed shaker for extraction. A 10-mL aliquot of the supernatant was filtered, evaporated to approximately 10% of its original volume, then re-filtered and evaporated to dryness. The samples were reconstituted in 0.10 mL of 25% methanol in water, yielding a 100-fold concentration factor. Dicamba was quantitated using LC-MS/MS with electrospray ionization in negative ion mode with SelexION<sup>®</sup>+ Differential Mobility Separation. The working range of the method was from 0.030 to 7.5 ng/PUF (0.100 to 25.0 ng/mL injected on column), with an LOQ of 0.10 ng/PUF. Analysis conditions are presented in [Appendix M](#).

##### 4.3.2 On-going Method Performance Verification

The analytical method was validated for PUF at Eurofins, Columbia, MO, and the LOQ was demonstrated to be 0.10 ng/PUF ([Rebstock, 2019](#)). Concurrent fortifications within this study performed by Eurofins met accuracy, precision, and selectivity criteria ([Table 5](#)). These data confirm the performance of the method with a LOQ of 0.10 ng/PUF as defined in ME-2242 ([Appendix D](#)).

##### 4.3.3 PUF Sample Preparation and Storage

The PUF samples were received frozen and were maintained frozen in a continuously monitored freezer set at approximately -20 °C until removed for extraction. All field collected PUF samples in this study were extracted within a period of 15 days after collection ([Appendix H](#)). All field exposed QC and transit stability PUF samples were extracted within a period of 17 days after fortification. Stability of dicamba on PUF collectors has been demonstrated for at least 78 days during frozen storage in a stability study ([Maher, 2016](#)). The stability of the residues during analysis (from extraction to instrumental analysis) has been demonstrated for at least 7 days during refrigerated storage ([Rebstock, 2019](#)). All PUF samples were analyzed within two (2) days after extraction, which is within demonstrated stability.

##### 4.3.4 PUF Sample Analysis

The samples were extracted and analyzed starting on July 15, 2019. Dates of sample extraction and analysis are provided in the significant dates table ([Appendix H](#)).

Control PUF samples were fortified with dicamba standard for determination of the method recovery. Controls and fortified controls were analyzed concurrently with the field samples. The averages and ranges of recoveries from laboratory-fortified PUF samples are summarized in [Table 5](#). Detailed recovery results can be found in [Appendix G](#).

#### 5. ANALYTICAL DEVIATIONS

A method deviation dated September 04, 2019 documented one instance during the study on which the percent relative standard deviation (RSD) of LOQ quality control (QC) fortification recoveries within an analytical set following analytical method ME-2242 was >20% RSD.

A Eurofins SOP deviation dated August 27, 2019 documented one instance during the study on which the display settings for the pipette used to prepare the transit stability and field QC samples was not documented.

None of the deviations described above were determined to have had a significant negative impact on the integrity of the study.

## **6. RESULTS**

### **6.1 Application Monitoring Filter Paper Sample Results**

Residues of dicamba on filter papers that were placed in the field plots to verify that the target dicamba application rates were sprayed were determined by ME-2166 ([Appendix B](#)). The average recoveries by fortification level were 104% (target 0.516 mg dicamba/filter) and 103% (target 0.860 mg dicamba/filter). A summary of recoveries from laboratory prepared quality check filter paper samples is located in [Table 1](#). A summary of the analytical results of application monitoring filter paper samples is presented in [Table 2](#). The mean assay result was 0.631 mg dicamba/filter, and the theoretical application rate was 0.688 mg dicamba/filter, based on the nominal 0.5 lb dicamba a.e./acre application rate. The assay result was 92% of the theoretical application rate.

Detailed analytical results for application monitoring filter paper samples, including recovery data and statistics, are reported in [Appendix E](#). Representative standard chromatograms and calibration curve can be found in [Appendix I](#), and representative sample chromatograms can be found in [Appendix J](#).

### **6.2 Tank Mix Sample Results**

The tank mix samples were collected at two time points: 1) after the tank mix of the test substances had been fully mixed prior to the application, and 2) following application.

Tank mix sample results are reported as dicamba weight percent (wt%) and were determined by ME-2154 ([Appendix C](#)) with modifications. The average recoveries by fortification level were 99% (target 0.30 wt% dicamba) and 99% (target 0.50 wt% dicamba). A summary of recoveries from laboratory prepared quality check tank mix samples is located in [Table 3](#). A summary of the analytical results for tank mix samples is presented in [Table 4](#). Average wt% was 0.381% for pre-application samples and 0.380% for post-application samples, and the theoretical tank mix concentration was 0.40% dicamba based on the nominal 0.5 lb dicamba a.e./acre application rate. The assay results were 95% of the theoretical application rate for the pre-application and post-application samples.

Detailed analytical results for tank mix samples, including recovery data and statistics, are reported in [Appendix F](#). Representative standard chromatograms and calibration curve can be found in [Appendix I](#), and representative sample chromatograms can be found in [Appendix K](#).

### **6.3 PUF Sample Results**

#### **6.3.1 Procedural Recoveries**

Residues of dicamba in all PUF samples were quantified by LC-MS/MS. The performance of the analytical method was evaluated by fortifying controls with the reference substance.

The recovery of fortified procedural PUF samples were corrected for the average background control content within each analytical set. Acceptable recovery data were obtained for dicamba and are shown in [Table 5](#). Average recoveries by fortification level were 103% (0.10 ng/PUF), 99% (6.0 ng/PUF) and 98% (60 ng/PUF).

Detailed recovery results for PUF samples can be found in [Appendix G](#). A calibration curve and representative standard, control, and fortified control chromatograms can be found in [Appendix L](#).

### **6.3.2 Individual PUF Sample Results**

Results are reported as ng/PUF sample collector. While more decimal places may be displayed in raw data tables, the results are being reported with no more than three significant figures in summary tables. The residue values for unknown samples are not corrected for background control content. Summaries of the analytical data are presented in [Table 6](#) for center mast PUF samples and [Table 7](#) for perimeter PUF samples.

Detailed analytical results for PUF samples can be found in [Appendix G](#), and representative treated chromatograms can be found in [Appendix L](#).

### **6.3.3 Field Exposed and Transit Stability PUF Sample Results**

Field exposed spike samples were prepared in the lab at Eurofins, Columbia, MO. There were six (6) samples at each of the following levels: 0, 3, 10, and 30 ng dicamba/PUF. They were stored at -20°C, then they were shipped on dry ice overnight to the field site. Three samples at each level were weathered for approximately 6 and 12 hours, respectively, to determine the potential amount of dicamba lost during a sampling event. Weathering consisted of drawing air across the field exposed spikes. The samples were placed where no additional exposure to dicamba was expected to occur. The analytical results are summarized in [Table 8](#). Recoveries of the field exposed QC samples ranged from 89% to 109% with an average recovery of 98% across all fortification levels.

To mimic sample transportation from the field to Eurofins, three (3) PUF samples were fortified in the lab at 30 ng/PUF and placed on dry ice along with three untreated control PUF samples. They were stored at -20°C, then they were shipped on dry ice overnight to the field site where they were stored at approximately -20°C. These samples were designated as transit stability samples, and the analytical results are summarized in [Table 9](#). Recoveries of the transit stability samples ranged from 92% to 106% with an average recovery of 99%. Field exposed spike and transit stability samples were hand delivered to the analytical lab along with field samples after seven days.

Detailed analytical results for both field exposed spike and transit stability samples can be found in [Appendix G](#), and representative chromatograms for each are included in [Appendix L](#).

## **7. REFERENCES**

Rebstock, M. 2019. Development and Validation for the Analytical Method ME-2242: LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps. EAG Study 88605. Monsanto Technical Report MSL0030621. Columbia, Missouri.

Maher, D. 2016. Storage Stability of Dicamba on Polyurethane Foam Air Sampling Traps. Monsanto Technical Report MSL0026782. St. Louis, Missouri.

## **8. TABLES**

**Table 1. Recovery of Dicamba from Fortified Application Monitoring Filter Paper Samples**

<b>Target Fort. Amt. (mg/filter)</b>	<b>Average Recovery (%)</b>	<b>Number of Fortifications</b>	<b>Minimum Recovery (%)</b>	<b>Maximum Recovery (%)</b>	<b>Std. Dev. (%)</b>	<b>RSD<sup>1</sup> (%)</b>
0.516	104	3	103	104	0.6	0.6
0.860	103	3	103	103	0.0	0.0

<sup>1</sup> The relative standard deviation (RSD) is calculated as the standard deviation divided by average % recovery.

**Table 2. Application Monitoring Filter Paper Sample Results**

Sample ID	Total Dicamba (mg/filter)	Percent of Target (%) <sup>1</sup>
REG-2019-0035 . FPS . A . APP . 1 . 003	0.677	98
REG-2019-0035 . FPS . A . APP . 2 . 004	0.729	106
REG-2019-0035 . FPS . A . APP . 3 . 005	0.732	106
REG-2019-0035 . FPS . A . APP . 4 . 006	0.650	94
REG-2019-0035 . FPS . B . APP . 1 . 007	0.660	96
REG-2019-0035 . FPS . B . APP . 2 . 008	0.579	84
REG-2019-0035 . FPS . B . APP . 3 . 009	0.676	98
REG-2019-0035 . FPS . B . APP . 4 . 010	0.644	94
REG-2019-0035 . FPS . C . APP . 1 . 011	0.619	90
REG-2019-0035 . FPS . C . APP . 2 . 012	0.647	94
REG-2019-0035 . FPS . C . APP . 3 . 013	0.508	74
REG-2019-0035 . FPS . C . APP . 4 . 014	0.554	81
REG-2019-0035 . FPS . D . APP . 1 . 015	0.603	88
REG-2019-0035 . FPS . D . APP . 2 . 016	0.621	90
REG-2019-0035 . FPS . D . APP . 3 . 017	0.625	91
REG-2019-0035 . FPS . D . APP . 4 . 018	0.571	83
<b>Average Amount of Dicamba (mg/filter)</b>	<b>0.631</b>	<b>92</b>
<b>Standard Deviation</b>	<b>0.060</b>	<b>8.6</b>

<sup>1</sup> Percent of the theoretical target rate based on a 0.5 lb dicamba a.e./acre (0.688 mg/filter)



**Table 3. Recovery of Dicamba from Fortified Tank Mix Samples**

<b>Target Fort. Amt. (wt %)</b>	<b>Average Recovery (%)</b>	<b>Number of Fortifications</b>	<b>Minimum Recovery (%)</b>	<b>Maximum Recovery (%)</b>	<b>Std. Dev. (%)</b>	<b>RSD<sup>1</sup> (%)</b>
0.30	99	3	99	99	0.0	0.0
0.50	99	3	99	99	0.0	0.0

<sup>1</sup> The relative standard deviation (RSD) is calculated as the standard deviation divided by average % recovery.

**Table 4. Tank Mix Sample Results**

Sample Description	Sample ID	pH	Dicamba (wt %)	Mean (wt %)	RSD (%) <sup>1</sup>	Percent of Target (%) <sup>2</sup>
Tank Mix Before Application	REG-2019-0035 . TNK . A . PRE-APP . 1 . 019	5.2	0.380	0.381	0.20	95
	REG-2019-0035 . TNK . A . PRE-APP . 2 . 020	5.2	0.381			
	REG-2019-0035 . TNK . A . PRE-APP . 3 . 021	5.2	0.382			
Tank Mix After Application	REG-2019-0035 . TNK . A . POST-APP . 1 . 022	5.2	0.380	0.380	0.10	95
	REG-2019-0035 . TNK . A . POST-APP . 2 . 023	5.2	0.380			
	REG-2019-0035 . TNK . A . POST-APP . 3 . 024	5.2	0.380			

<sup>1</sup> The relative standard deviation (RSD) is calculated as the standard deviation divided by average % recovery.

<sup>2</sup> Percent of the theoretical target rate based on a 0.5 lb dicamba a.e./acre (0.40 wt% dicamba)

**Table 5. Recovery of Dicamba from Fortified Polyurethane Foam Samples**

<b>Fort. Amt. (ng/PUF)</b>	<b>Average Recovery<sup>1</sup> (%)</b>	<b>Number of Fortifications</b>	<b>Minimum Recovery (%)</b>	<b>Maximum Recovery (%)</b>	<b>Std. Dev. (%)</b>	<b>RSD<sup>2</sup> (%)</b>
0.100	103	15	78	128	12.5	12.1
6.00	99	15	94	105	3.8	3.9
60.0	98	3	96	101	2.5	2.6

<sup>1</sup> The recoveries are corrected for apparent residues in the corresponding control samples

<sup>2</sup> The relative standard deviation (RSD) is calculated as the standard deviation divided by average % recovery.

**Table 6. Center Mast PUF Sample Results**

Sample ID	Sampling Time	Sample Location/Height	Dicamba (ng/PUF) <sup>1</sup>
REG-2019-0035 . PCM . 015 . PRE . 1 . 001	Pre-application	Center Mast 0.15 m	0.0761*
REG-2019-0035 . PCM . 015 . PRE . 2 . 002	Pre-application	Center Mast 0.15 m	0.0778*
REG-2019-0035 . PCM . 015 . 0-6 hr . 1 . 025	0-6 hours	Center Mast 0.15 m	8.34
REG-2019-0035 . PCM . 033 . 0-6 hr . 1 . 026	0-6 hours	Center Mast 0.33 m	6.82
REG-2019-0035 . PCM . 055 . 0-6 hr . 1 . 027	0-6 hours	Center Mast 0.55 m	4.64
REG-2019-0035 . PCM . 090 . 0-6 hr . 1 . 028	0-6 hours	Center Mast 0.90 m	3.35
REG-2019-0035 . PCM . 150 . 0-6 hr . 1 . 029	0-6 hours	Center Mast 1.5 m	3.04
REG-2019-0035 . PCM . 150 . 0-6 hr . 2 . 030	0-6 hours	Center Mast 1.5 m	2.57
REG-2019-0035 . PCM . 015 . 6-12 . 1 . 039	6-12 hours	Center Mast 0.15 m	0.908
REG-2019-0035 . PCM . 033 . 6-12 . 1 . 040	6-12 hours	Center Mast 0.33 m	0.925
REG-2019-0035 . PCM . 055 . 6-12 . 1 . 041	6-12 hours	Center Mast 0.55 m	0.549
REG-2019-0035 . PCM . 090 . 6-12 . 1 . 042	6-12 hours	Center Mast 0.90 m	0.573
REG-2019-0035 . PCM . 150 . 6-12 . 1 . 043	6-12 hours	Center Mast 1.5 m	0.397
REG-2019-0035 . PCM . 150 . 6-12 . 2 . 044	6-12 hours	Center Mast 1.5 m	0.416
REG-2019-0035 . PCM . 015 . 12-24 hr . 1 . 053	12-24 hours	Center Mast 0.15 m	2.30
REG-2019-0035 . PCM . 033 . 12-24 hr . 1 . 054	12-24 hours	Center Mast 0.33 m	2.12
REG-2019-0035 . PCM . 055 . 12-24 hr . 1 . 055	12-24 hours	Center Mast 0.55 m	2.64
REG-2019-0035 . PCM . 090 . 12-24 hr . 1 . 056	12-24 hours	Center Mast 0.90 m	ND
REG-2019-0035 . PCM . 150 . 12-24 hr . 1 . 057	12-24 hours	Center Mast 1.5 m	1.07
REG-2019-0035 . PCM . 150 . 12-24 hr . 2 . 058	12-24 hours	Center Mast 1.5 m	1.20
REG-2019-0035 . PCM . 015 . 24-36 hr . 1 . 067	24-36 hours	Center Mast 0.15 m	1.89
REG-2019-0035 . PCM . 033 . 24-36 hr . 1 . 068	24-36 hours	Center Mast 0.33 m	1.29
REG-2019-0035 . PCM . 055 . 24-36 hr . 1 . 069	24-36 hours	Center Mast 0.55 m	1.22
REG-2019-0035 . PCM . 090 . 24-36 hr . 1 . 070	24-36 hours	Center Mast 0.90 m	0.952
REG-2019-0035 . PCM . 150 . 24-36 hr . 1 . 071	24-36 hours	Center Mast 1.5 m	1.02
REG-2019-0035 . PCM . 150 . 24-36 hr . 2 . 072	24-36 hours	Center Mast 1.5 m	0.826
REG-2019-0035 . PCM . 015 . 36-48 hr . 1 . 081	36-48 hours	Center Mast 0.15 m	5.24
REG-2019-0035 . PCM . 033 . 36-48 hr . 1 . 082	36-48 hours	Center Mast 0.33 m	2.02
REG-2019-0035 . PCM . 055 . 36-48 hr . 1 . 083	36-48 hours	Center Mast 0.55 m	1.01
REG-2019-0035 . PCM . 090 . 36-48 hr . 1 . 084	36-48 hours	Center Mast 0.90 m	0.575
REG-2019-0035 . PCM . 150 . 36-48 hr . 1 . 085	36-48 hours	Center Mast 1.5 m	0.468
REG-2019-0035 . PCM . 150 . 36-48 hr . 2 . 086	36-48 hours	Center Mast 1.5 m	0.487
REG-2019-0035 . PCM . 015 . 48-60 hr . 1 . 095	48-60 hours	Center Mast 0.15 m	0.568
REG-2019-0035 . PCM . 033 . 48-60 hr . 1 . 096	48-60 hours	Center Mast 0.33 m	0.567

<sup>1</sup> ND = no observable peak or observed residues back calculating to a value less than or equal to zero.  
Results greater than the LOD (defined as 30% of the LOQ or 0.03 ng/PUF) but less than the LOQ (0.1 ng/PUF) are marked with an \*.

**Table 6. Center Mast PUF Sample Results (continued)**

Sample ID	Sampling Time	Sample Location/Height	Dicamba (ng/PUF)
REG-2019-0035 . PCM . 055 . 48-60 hr . 1 . 097	48-60 hours	Center Mast 0.55 m	0.526
REG-2019-0035 . PCM . 090 . 48-60 hr . 1 . 098	48-60 hours	Center Mast 0.90 m	0.505
REG-2019-0035 . PCM . 150 . 48-60 hr . 1 . 099	48-60 hours	Center Mast 1.5 m	0.472
REG-2019-0035 . PCM . 150 . 48-60 hr . 2 . 100	48-60 hours	Center Mast 1.5 m	0.532
REG-2019-0035 . PCM . 015 . 60-72 hr . 1 . 109	60-72 hours	Center Mast 0.15 m	0.538
REG-2019-0035 . PCM . 033 . 60-72 hr . 1 . 110	60-72 hours	Center Mast 0.33 m	0.515
REG-2019-0035 . PCM . 055 . 60-72 hr . 1 . 111	60-72 hours	Center Mast 0.55 m	0.506
REG-2019-0035 . PCM . 090 . 60-72 hr . 1 . 112	60-72 hours	Center Mast 0.90 m	0.501
REG-2019-0035 . PCM . 150 . 60-72 hr . 1 . 113	60-72 hours	Center Mast 1.5 m	0.329
REG-2019-0035 . PCM . 150 . 60-72 hr . 2 . 114	60-72 hours	Center Mast 1.5 m	0.432
REG-2019-0035 . PCM . 015 . 72-84 hr . 1 . 123	72-84 hours	Center Mast 0.15 m	0.345
REG-2019-0035 . PCM . 033 . 72-84 hr . 1 . 124	72-84 hours	Center Mast 0.33 m	0.298
REG-2019-0035 . PCM . 055 . 72-84 hr . 1 . 125	72-84 hours	Center Mast 0.55 m	0.265
REG-2019-0035 . PCM . 090 . 72-84 hr . 1 . 126	72-84 hours	Center Mast 0.90 m	0.269
REG-2019-0035 . PCM . 150 . 72-84 hr . 1 . 127	72-84 hours	Center Mast 1.5 m	0.262
REG-2019-0035 . PCM . 150 . 72-84 hr . 2 . 128	72-84 hours	Center Mast 1.5 m	0.232
REG-2019-0035 . PCM . 015 . 84-96 hr . 1 . 137	84-96 hours	Center Mast 0.15 m	1.01
REG-2019-0035 . PCM . 033 . 84-96 hr . 1 . 138	84-96 hours	Center Mast 0.33 m	1.46
REG-2019-0035 . PCM . 055 . 84-96 hr . 1 . 139	84-96 hours	Center Mast 0.55 m	0.235
REG-2019-0035 . PCM . 090 . 84-96 hr . 1 . 140	84-96 hours	Center Mast 0.90 m	0.196
REG-2019-0035 . PCM . 150 . 84-96 hr . 1 . 141	84-96 hours	Center Mast 1.5 m	0.115
REG-2019-0035 . PCM . 150 . 84-96 hr . 2 . 142	84-96 hours	Center Mast 1.5 m	0.132
REG-2019-0035 . PCM . 015 . 96-108 hr . 1 . 151	96-108 hours	Center Mast 0.15 m	0.885
REG-2019-0035 . PCM . 033 . 96-108 hr . 1 . 152	96-108 hours	Center Mast 0.33 m	0.634
REG-2019-0035 . PCM . 055 . 96-108 hr . 1 . 153	96-108 hours	Center Mast 0.55 m	0.205
REG-2019-0035 . PCM . 090 . 96-108 hr . 1 . 154	96-108 hours	Center Mast 0.90 m	0.154
REG-2019-0035 . PCM . 150 . 96-108 hr . 1 . 155	96-108 hours	Center Mast 1.5 m	0.288
REG-2019-0035 . PCM . 150 . 96-108 hr . 2 . 156	96-108 hours	Center Mast 1.5 m	0.203
REG-2019-0035 . PCM . 015 . 108-120 hr . 1 . 165	108-120 hours	Center Mast 0.15 m	0.319
REG-2019-0035 . PCM . 033 . 108-120 hr . 1 . 166	108-120 hours	Center Mast 0.33 m	0.264
REG-2019-0035 . PCM . 055 . 108-120 hr . 1 . 167	108-120 hours	Center Mast 0.55 m	0.378
REG-2019-0035 . PCM . 090 . 108-120 hr . 1 . 168	108-120 hours	Center Mast 0.90 m	0.296
REG-2019-0035 . PCM . 150 . 108-120 hr . 1 . 169	108-120 hours	Center Mast 1.5 m	0.331
REG-2019-0035 . PCM . 150 . 108-120 hr . 2 . 170	108-120 hours	Center Mast 1.5 m	0.269

**Table 6. Center Mast PUF Sample Results (continued)**

Sample ID	Sampling Time	Sample Location/Height	Dicamba (ng/PUF)
REG-2019-0035 . PCM . 015 . 120-132 hr . 1 . 179	120-132 hours	Center Mast 0.15 m	0.336
REG-2019-0035 . PCM . 033 . 120-132 hr . 1 . 180	120-132 hours	Center Mast 0.33 m	0.438
REG-2019-0035 . PCM . 055 . 120-132 hr . 1 . 181	120-132 hours	Center Mast 0.55 m	0.542
REG-2019-0035 . PCM . 090 . 120-132 hr . 1 . 182	120-132 hours	Center Mast 0.90 m	0.283
REG-2019-0035 . PCM . 150 . 120-132 hr . 1 . 183	120-132 hours	Center Mast 1.5 m	0.389
REG-2019-0035 . PCM . 150 . 120-132 hr . 2 . 184	120-132 hours	Center Mast 1.5 m	0.339
REG-2019-0035 . PCM . 015 . 132-144 hr . 1 . 193	132-144 hours	Center Mast 0.15 m	0.528
REG-2019-0035 . PCM . 033 . 132-144 hr . 1 . 194	132-144 hours	Center Mast 0.33 m	0.571
REG-2019-0035 . PCM . 055 . 132-144 hr . 1 . 195	132-144 hours	Center Mast 0.55 m	0.572
REG-2019-0035 . PCM . 090 . 132-144 hr . 1 . 196	132-144 hours	Center Mast 0.90 m	0.599
REG-2019-0035 . PCM . 150 . 132-144 hr . 1 . 197	132-144 hours	Center Mast 1.5 m	0.580
REG-2019-0035 . PCM . 150 . 132-144 hr . 2 . 198	132-144 hours	Center Mast 1.5 m	0.649
REG-2019-0035 . PCM . 015 . 144-156 hr . 1 . 207	144-156 hours	Center Mast 0.15 m	0.305
REG-2019-0035 . PCM . 033 . 144-156 hr . 1 . 208	144-156 hours	Center Mast 0.33 m	2.89
REG-2019-0035 . PCM . 055 . 144-156 hr . 1 . 209	144-156 hours	Center Mast 0.55 m	0.396
REG-2019-0035 . PCM . 090 . 144-156 hr . 1 . 210	144-156 hours	Center Mast 0.90 m	0.309
REG-2019-0035 . PCM . 150 . 144-156 hr . 1 . 211	144-156 hours	Center Mast 1.5 m	0.757
REG-2019-0035 . PCM . 150 . 144-156 hr . 2 . 212	144-156 hours	Center Mast 1.5 m	0.380
REG-2019-0035 . PCM . 015 . 156-168 hr . 1 . 221	156-168 hours	Center Mast 0.15 m	0.833
REG-2019-0035 . PCM . 033 . 156-168 hr . 1 . 222	156-168 hours	Center Mast 0.33 m	0.921
REG-2019-0035 . PCM . 055 . 156-168 hr . 1 . 223	156-168 hours	Center Mast 0.55 m	1.03
REG-2019-0035 . PCM . 090 . 156-168 hr . 1 . 224	156-168 hours	Center Mast 0.90 m	1.02
REG-2019-0035 . PCM . 150 . 156-168 hr . 1 . 225	156-168 hours	Center Mast 1.5 m	1.42
REG-2019-0035 . PCM . 150 . 156-168 hr . 2 . 226	156-168 hours	Center Mast 1.5 m	1.11

**Table 7. Perimeter PUF Sample Results**

Sample ID	Sampling Time	Sample Location	Dicamba (ng/PUF)
REG-2019-0035 . POF . A . 0-6 hr . 1 . 031	0-6 hours	Perimeter Transect A	2.13
REG-2019-0035 . POF . B . 0-6 hr . 1 . 032	0-6 hours	Perimeter Transect B	1.22
REG-2019-0035 . POF . C . 0-6 hr . 1 . 033	0-6 hours	Perimeter Transect C	2.57
REG-2019-0035 . POF . D . 0-6 hr . 1 . 034	0-6 hours	Perimeter Transect D	1.52
REG-2019-0035 . POF . E . 0-6 hr . 1 . 035	0-6 hours	Perimeter Transect E	1.14
REG-2019-0035 . POF . F . 0-6 hr . 1 . 036	0-6 hours	Perimeter Transect F	0.138
REG-2019-0035 . POF . G . 0-6 hr . 1 . 037	0-6 hours	Perimeter Transect G	0.172
REG-2019-0035 . POF . H . 0-6 hr . 1 . 038	0-6 hours	Perimeter Transect H	0.317
REG-2019-0035 . POF . A . 6-12 hr . 1 . 045	6-12 hours	Perimeter Transect A	0.371
REG-2019-0035 . POF . B . 6-12 hr . 1 . 046	6-12 hours	Perimeter Transect B	0.290
REG-2019-0035 . POF . C . 6-12 hr . 1 . 047	6-12 hours	Perimeter Transect C	0.339
REG-2019-0035 . POF . D . 6-12 hr . 1 . 048	6-12 hours	Perimeter Transect D	0.169
REG-2019-0035 . POF . E . 6-12 hr . 1 . 049	6-12 hours	Perimeter Transect E	0.165
REG-2019-0035 . POF . F . 6-12 hr . 1 . 050	6-12 hours	Perimeter Transect F	0.265
REG-2019-0035 . POF . G . 6-12 hr . 1 . 051	6-12 hours	Perimeter Transect G	0.226
REG-2019-0035 . POF . H . 6-12 hr . 1 . 052	6-12 hours	Perimeter Transect H	0.121
REG-2019-0035 . POF . A . 12-24 hr . 1 . 059	12-24 hours	Perimeter Transect A	1.58
REG-2019-0035 . POF . B . 12-24 hr . 1 . 060	12-24 hours	Perimeter Transect B	1.10
REG-2019-0035 . POF . C . 12-24 hr . 1 . 061	12-24 hours	Perimeter Transect C	3.29
REG-2019-0035 . POF . D . 12-24 hr . 1 . 062	12-24 hours	Perimeter Transect D	0.626
REG-2019-0035 . POF . E . 12-24 hr . 1 . 063	12-24 hours	Perimeter Transect E	0.854
REG-2019-0035 . POF . F . 12-24 hr . 1 . 064	12-24 hours	Perimeter Transect F	0.755
REG-2019-0035 . POF . G . 12-24 hr . 1 . 065	12-24 hours	Perimeter Transect G	0.759
REG-2019-0035 . POF . H . 12-24 hr . 1 . 066	12-24 hours	Perimeter Transect H	0.780
REG-2019-0035 . POF . A . 24-36 hr . 1 . 073	24-36 hours	Perimeter Transect A	0.396
REG-2019-0035 . POF . B . 24-36 hr . 1 . 074	24-36 hours	Perimeter Transect B	0.467
REG-2019-0035 . POF . C . 24-36 hr . 1 . 075	24-36 hours	Perimeter Transect C	1.66
REG-2019-0035 . POF . D . 24-36 hr . 1 . 076	24-36 hours	Perimeter Transect D	0.347
REG-2019-0035 . POF . E . 24-36 hr . 1 . 077	24-36 hours	Perimeter Transect E	0.623
REG-2019-0035 . POF . F . 24-36 hr . 1 . 078	24-36 hours	Perimeter Transect F	0.185
REG-2019-0035 . POF . G . 24-36 hr . 1 . 079	24-36 hours	Perimeter Transect G	0.167
REG-2019-0035 . POF . H . 24-36 hr . 1 . 080	24-36 hours	Perimeter Transect H	0.189
REG-2019-0035 . POF . A . 36-48 hr . 1 . 087	36-48 hours	Perimeter Transect A	0.300
REG-2019-0035 . POF . B . 36-48 hr . 1 . 088	36-48 hours	Perimeter Transect B	0.355

**Table 7. Perimeter PUF Sample Results (continued)**

Sample ID	Sampling Time	Sample Location	Dicamba (ng/PUF) <sup>1</sup>
REG-2019-0035 . POF . C . 36-48 hr . 1 . 089	36-48 hours	Perimeter Transect C	8.33
REG-2019-0035 . POF . D . 36-48 hr . 1 . 090	36-48 hours	Perimeter Transect D	0.198
REG-2019-0035 . POF . E . 36-48 hr . 1 . 091	36-48 hours	Perimeter Transect E	0.311
REG-2019-0035 . POF . F . 36-48 hr . 1 . 092	36-48 hours	Perimeter Transect F	0.341
REG-2019-0035 . POF . G . 36-48 hr . 1 . 093	36-48 hours	Perimeter Transect G	0.982
REG-2019-0035 . POF . H . 36-48 hr . 1 . 094	36-48 hours	Perimeter Transect H	0.418
REG-2019-0035 . POF . A . 48-60 hr . 1 . 101	48-60 hours	Perimeter Transect A	0.520
REG-2019-0035 . POF . B . 48-60 hr . 1 . 102	48-60 hours	Perimeter Transect B	0.418
REG-2019-0035 . POF . C . 48-60 hr . 1 . 103	48-60 hours	Perimeter Transect C	2.11
REG-2019-0035 . POF . D . 48-60 hr . 1 . 104	48-60 hours	Perimeter Transect D	0.407
REG-2019-0035 . POF . E . 48-60 hr . 1 . 105	48-60 hours	Perimeter Transect E	0.523
REG-2019-0035 . POF . F . 48-60 hr . 1 . 106	48-60 hours	Perimeter Transect F	0.490
REG-2019-0035 . POF . G . 48-60 hr . 1 . 107	48-60 hours	Perimeter Transect G	0.518
REG-2019-0035 . POF . H . 48-60 hr . 1 . 108	48-60 hours	Perimeter Transect H	0.444
REG-2019-0035 . POF . A . 60-72 hr . 1 . 115	60-72 hours	Perimeter Transect A	0.381
REG-2019-0035 . POF . B . 60-72 hr . 1 . 116	60-72 hours	Perimeter Transect B	0.375
REG-2019-0035 . POF . C . 60-72 hr . 1 . 117	60-72 hours	Perimeter Transect C	0.447
REG-2019-0035 . POF . D . 60-72 hr . 1 . 118	60-72 hours	Perimeter Transect D	0.460
REG-2019-0035 . POF . E . 60-72 hr . 1 . 119	60-72 hours	Perimeter Transect E	0.557
REG-2019-0035 . POF . F . 60-72 hr . 1 . 120	60-72 hours	Perimeter Transect F	0.541
REG-2019-0035 . POF . G . 60-72 hr . 1 . 121	60-72 hours	Perimeter Transect G	1.33
REG-2019-0035 . POF . H . 60-72 hr . 1 . 122	60-72 hours	Perimeter Transect H	0.482
REG-2019-0035 . POF . A . 72-84 hr . 1 . 129	72-84 hours	Perimeter Transect A	0.262
REG-2019-0035 . POF . B . 72-84 hr . 1 . 130	72-84 hours	Perimeter Transect B	0.264
REG-2019-0035 . POF . C . 72-84 hr . 1 . 131	72-84 hours	Perimeter Transect C	0.330
REG-2019-0035 . POF . D . 72-84 hr . 1 . 132	72-84 hours	Perimeter Transect D	0.210
REG-2019-0035 . POF . E . 72-84 hr . 1 . 133	72-84 hours	Perimeter Transect E	0.250
REG-2019-0035 . POF . F . 72-84 hr . 1 . 134	72-84 hours	Perimeter Transect F	0.198
REG-2019-0035 . POF . G . 72-84 hr . 1 . 135	72-84 hours	Perimeter Transect G	0.214
REG-2019-0035 . POF . H . 72-84 hr . 1 . 136	72-84 hours	Perimeter Transect H	0.228
REG-2019-0035 . POF . A . 84-96 hr . 1 . 143	84-96 hours	Perimeter Transect A	0.0851*
REG-2019-0035 . POF . B . 84-96 hr . 1 . 144	84-96 hours	Perimeter Transect B	0.124
REG-2019-0035 . POF . C . 84-96 hr . 1 . 145	84-96 hours	Perimeter Transect C	0.167
REG-2019-0035 . POF . D . 84-96 hr . 1 . 146	84-96 hours	Perimeter Transect D	0.231

<sup>1</sup> Results greater than the LOD (defined as 30% of the LOQ or 0.03 ng/PUF) but less than the LOQ (0.1 ng/PUF) are marked with an \*.



**Table 7. Perimeter PUF Sample Results (continued)**

Sample ID	Sampling Time	Sample Location	Dicamba (ng/PUF)
REG-2019-0035 . POF . E . 84-96 hr . 1 . 147	84-96 hours	Perimeter Transect E	0.127
REG-2019-0035 . POF . F . 84-96 hr . 1 . 148	84-96 hours	Perimeter Transect F	0.122
REG-2019-0035 . POF . G . 84-96 hr . 1 . 149	84-96 hours	Perimeter Transect G	0.151
REG-2019-0035 . POF . H . 84-96 hr . 1 . 150	84-96 hours	Perimeter Transect H	0.105
REG-2019-0035 . POF . A . 96-108 hr . 1 . 157	96-108 hours	Perimeter Transect A	0.117
REG-2019-0035 . POF . B . 96-108 hr . 1 . 158	96-108 hours	Perimeter Transect B	0.106
REG-2019-0035 . POF . C . 96-108 hr . 1 . 159	96-108 hours	Perimeter Transect C	0.345
REG-2019-0035 . POF . D . 96-108 hr . 1 . 160	96-108 hours	Perimeter Transect D	0.183
REG-2019-0035 . POF . E . 96-108 hr . 1 . 161	96-108 hours	Perimeter Transect E	0.181
REG-2019-0035 . POF . F . 96-108 hr . 1 . 162	96-108 hours	Perimeter Transect F	0.120
REG-2019-0035 . POF . G . 96-108 hr . 1 . 163	96-108 hours	Perimeter Transect G	0.192
REG-2019-0035 . POF . H . 96-108 hr . 1 . 164	96-108 hours	Perimeter Transect H	0.123
REG-2019-0035 . POF . A . 108-120 hr . 1 . 171	108-120 hours	Perimeter Transect A	0.994
REG-2019-0035 . POF . B . 108-120 hr . 1 . 172	108-120 hours	Perimeter Transect B	0.290
REG-2019-0035 . POF . C . 108-120 hr . 1 . 173	108-120 hours	Perimeter Transect C	0.338
REG-2019-0035 . POF . D . 108-120 hr . 1 . 174	108-120 hours	Perimeter Transect D	0.256
REG-2019-0035 . POF . E . 108-120 hr . 1 . 175	108-120 hours	Perimeter Transect E	0.247
REG-2019-0035 . POF . F . 108-120 hr . 1 . 176	108-120 hours	Perimeter Transect F	0.234
REG-2019-0035 . POF . G . 108-120 hr . 1 . 177	108-120 hours	Perimeter Transect G	0.269
REG-2019-0035 . POF . H . 108-120 hr . 1 . 178	108-120 hours	Perimeter Transect H	0.268
REG-2019-0035 . POF . A . 120-132 hr . 1 . 185	120-132 hours	Perimeter Transect A	0.426
REG-2019-0035 . POF . B . 120-132 hr . 1 . 186	120-132 hours	Perimeter Transect B	0.266
REG-2019-0035 . POF . C . 120-132 hr . 1 . 187	120-132 hours	Perimeter Transect C	0.657
REG-2019-0035 . POF . D . 120-132 hr . 1 . 188	120-132 hours	Perimeter Transect D	0.256
REG-2019-0035 . POF . E . 120-132 hr . 1 . 189	120-132 hours	Perimeter Transect E	0.260
REG-2019-0035 . POF . F . 120-132 hr . 1 . 190	120-132 hours	Perimeter Transect F	0.219
REG-2019-0035 . POF . G . 120-132 hr . 1 . 191	120-132 hours	Perimeter Transect G	0.211
REG-2019-0035 . POF . H . 120-132 hr . 1 . 192	120-132 hours	Perimeter Transect H	0.360
REG-2019-0035 . POF . A . 132-144 hr . 1 . 199	132-144 hours	Perimeter Transect A	0.644
REG-2019-0035 . POF . B . 132-144 hr . 1 . 200	132-144 hours	Perimeter Transect B	0.651
REG-2019-0035 . POF . C . 132-144 hr . 1 . 201	132-144 hours	Perimeter Transect C	0.563
REG-2019-0035 . POF . D . 132-144 hr . 1 . 202	132-144 hours	Perimeter Transect D	0.501
REG-2019-0035 . POF . E . 132-144 hr . 1 . 203	132-144 hours	Perimeter Transect E	0.539
REG-2019-0035 . POF . F . 132-144 hr . 1 . 204	132-144 hours	Perimeter Transect F	0.426

**Table 7. Perimeter PUF Sample Results (continued)**

Sample ID	Sampling Time	Sample Location	Dicamba (ng/PUF)
REG-2019-0035 . POF . G . 132-144 hr . 1 . 205	132-144 hours	Perimeter Transect G	0.427
REG-2019-0035 . POF . H . 132-144 hr . 1 . 206	132-144 hours	Perimeter Transect H	0.614
REG-2019-0035 . POF . A . 144-156 hr . 1 . 213	144-156 hours	Perimeter Transect A	0.409
REG-2019-0035 . POF . B . 144-156 hr . 1 . 214	144-156 hours	Perimeter Transect B	0.295
REG-2019-0035 . POF . C . 144-156 hr . 1 . 215	144-156 hours	Perimeter Transect C	0.406
REG-2019-0035 . POF . D . 144-156 hr . 1 . 216	144-156 hours	Perimeter Transect D	0.313
REG-2019-0035 . POF . E . 144-156 hr . 1 . 217	144-156 hours	Perimeter Transect E	0.362
REG-2019-0035 . POF . F . 144-156 hr . 1 . 218	144-156 hours	Perimeter Transect F	0.368
REG-2019-0035 . POF . G . 144-156 hr . 1 . 219	144-156 hours	Perimeter Transect G	0.351
REG-2019-0035 . POF . H . 144-156 hr . 1 . 220	144-156 hours	Perimeter Transect H	0.633
REG-2019-0035 . POF . A . 156-168 hr . 1 . 227	156-168 hours	Perimeter Transect A	1.65
REG-2019-0035 . POF . B . 156-168 hr . 1 . 228	156-168 hours	Perimeter Transect B	1.26
REG-2019-0035 . POF . C . 156-168 hr . 1 . 229	156-168 hours	Perimeter Transect C	1.08
REG-2019-0035 . POF . D . 156-168 hr . 1 . 230	156-168 hours	Perimeter Transect D	0.966
REG-2019-0035 . POF . E . 156-168 hr . 1 . 231	156-168 hours	Perimeter Transect E	0.818
REG-2019-0035 . POF . F . 156-168 hr . 1 . 232	156-168 hours	Perimeter Transect F	0.829
REG-2019-0035 . POF . G . 156-168 hr . 1 . 233	156-168 hours	Perimeter Transect G	0.840
REG-2019-0035 . POF . H . 156-168 hr . 1 . 234	156-168 hours	Perimeter Transect H	0.866

**Table 8. Field Exposed PUF Sample Results**

Sample ID	Sampling Time	Fortification Level (ng/PUF)	Dicamba (ng/PUF) <sup>1</sup>	Percent Recovery (%)
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 1	6 hours	0	0.0436	NA
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 2	6 hours	0	0.121	NA
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 3	6 hours	0	0.0321	NA
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 1	6 hours	3	2.88	96
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 2	6 hours	3	2.88	96
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 3	6 hours	3	2.83	94
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 1	6 hours	10	9.34	93
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 2	6 hours	10	9.83	98
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 3	6 hours	10	9.54	95
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 1	6 hours	30	28.1	94
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 2	6 hours	30	32.8	109
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 3	6 hours	30	32.3	108
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 1	12 hours	0	<LOD	NA
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 2	12 hours	0	0.0523	NA
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 3	12 hours	0	<LOD	NA
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 1	12 hours	3	3.22	107
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 2	12 hours	3	2.96	99
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 3	12 hours	3	2.95	98
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 1	12 hours	10	8.91	89
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 2	12 hours	10	10.4	104
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 3	12 hours	10	9.25	93
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 1	12 hours	30	29.7	99
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 2	12 hours	30	29.8	99
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 3	12 hours	30	29.1	97

<sup>1</sup> <LOD = results greater than zero but less than the LOD (defined as 30% of the LOQ or 0.03 ng/PUF).

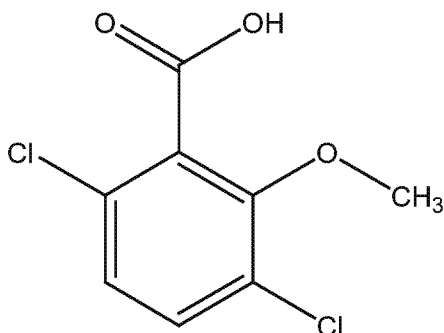
**Table 9. Transit Stability PUF Sample Results**

Sample ID	Fortification level (ng/PUF)	Dicamba (ng/PUF) <sup>1</sup>	Percent Recovery (%)
REG-2019-0035 . TSPUF . 0 ng . 1	0	ND	NA
REG-2019-0035 . TSPUF . 0 ng . 2	0	ND	NA
REG-2019-0035 . TSPUF . 0 ng . 3	0	ND	NA
REG-2019-0035 . TSPUF . 30 ng . 1	30	29.5	98
REG-2019-0035 . TSPUF . 30 ng . 2	30	27.6	92
REG-2019-0035 . TSPUF . 30 ng . 3	30	31.7	106

<sup>1</sup> ND = no observable peak or observed residues back calculating to a value less than or equal to zero.

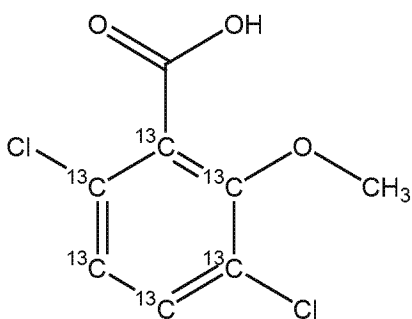
## 9. FIGURES

**Figure 1. Chemical Structure of Reference Substance**



Dicamba  
3,6-dichloro-2-methoxybenzoic acid

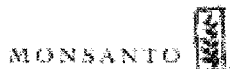
**Figure 2. Chemical Structure of Internal Standard**



<sup>13</sup>C<sub>6</sub>-dicamba  
3,6-dichloro-2-methoxybenzoic-1,2,3,4,5,6-<sup>13</sup>C<sub>6</sub> acid

## 10. APPENDICES

### Appendix A. Certificates of Analysis for the Reference Substance and Internal Standard



#### Analytical Reference Standard Certificate of Analysis

800 N. Lindbergh Blvd.  
St. Louis, MO 63167  
314-694-1000

ARS Name: Dicamba

Class 1

Freezerworks ID: 109334

Orig. Certification Date: 11/02/2016

Expiration Date: 09/30/2020

Purity: 99.4 %

Storage Condition: Ambient Desiccated

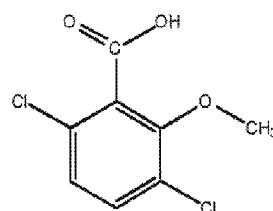
Molecular Formula:  $C_8H_6Cl_2O_3$

Molecular Weight: 221.04

Chemical Name: 3,6-dichloro-2-methoxybenzoic acid

Other Name(s): MON 11900, CAS# 1918-00-9

Structure:



#### Certification Tests

Appearance Off-White Solid

H-NMR Consistent with Structure

Moisture Analysis 0.54 %

Weight% Impurities 0 %

**Comments:** The (re)characterization of this standard showed no significant changes since the original certification. The expiration date has been extended for any sample of the same lot, if stored at the conditions indicated. This characterization was conducted in compliance with the requirements of the current version of Monsanto SOP PR-0522; as well as the United States EPA Federal Insecticide, Fungicide and Rodenticide Act (FIFRA); Good Laboratory Practice (GLP) Standards (40 CFR Part 160).

Certified By:

Jing Berry, ARSO

Date:

09/21/2018

Cert ID: 180711-22

Page 1 of 1



## Analytical Reference Standard Certificate of Analysis

800 N. Lindbergh Blvd.  
St. Louis, MO 63167  
314-694-1000

**Sample Name:** 13C Dicamba

**Class 5**

**Freezerworks ID:** 109335

**Structure:**

**Orig. Certification Date:** 10/04/2016

**Expiration Date:** 08/08/2021 Assigned by Cambridge Isotope Lab

**Purity:** Qualitative only. Purity not assigned.

**Appearance:** White Solid

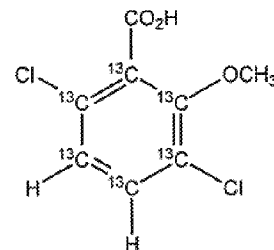
**Storage Condition:** Refrigerate

**Molecular Formula:**  $(^{12}\text{C})_2(^{13}\text{C})_6\text{H}_6\text{Cl}_2\text{O}_3$

**Molecular Weight:** 227.04

**Chemical Name:** 3,6-Dichloro-2-methoxybenzoic-1,2,3,4,5,6- $^{13}\text{C}_6$  acid

**Other Name(s):** CAS# 1173023-06-7



### Certification Tests

**Other** Vendor Certified

**Comments:** Isotopic Purity = 99.2 %

This characterization was conducted in compliance with the requirements of the current version of Monsanto SOP PR-0522.

**Certified By:**

Jing Berry, ARSO

**Date:**

07/23/2018

Cert ID: 160908-22

Page 1 of 1

ED 006453A 00002562-00188



## Appendix B. Analytical Method – Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

Monsanto Company Method

Effective Date: April 20, 2018      ME-2166-01      Page 1 of 9  
Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

### Overview

- Purpose & Scope** This document describes the method used by Environmental Sciences personnel to determine dicamba acid concentration after deposition onto filter paper.
- Method Summary** Dicamba acid is extracted from the filter paper using water. The sample tubes are capped and agitated on a high-speed shaker for extraction. An aliquot of the supernatant is filtered, and then dicamba is quantitated using HPLC-UV.
- Safety Precautions** Follow current safety policies. Important precautions include:
- Some solvents are volatile and/or flammable. Care must be taken to keep them away from any source of ignition.
  - Ensure proper ventilation to avoid excessive exposure to any toxic vapors.
  - Read and follow all safety warnings on reagent containers.
  - Ensure proper safety requirements are followed when operating equipment.

### Materials

**Equipment** The following equipment is used in this method. Specific brands are listed to aid the analyst in finding items. In most cases, equivalent equipment from other vendors may be used.

Equipment	Number/Specification
<b>Analytical Instruments</b>	
HPLC System	Gradient pump capable of pumping at 1.5 mL per minute, such as Agilent 1260 HPLC system, a UV detector, such as Agilent 1260 Infinity Variable Wavelength Detector and an autosampler capable of accurately and precisely injecting a defined volume
HPLC system	Agilent 1260 HPLC system: Solvent Degasser, at least 2 Pumps, Autosampler, Heated Column Compartment and Controller
HPLC column	HiChrom Alltima C18, 250 mm × 4.6 mm, 5 µm
<b>Sample Preparation Equipment</b>	
Analytical balance	Capable of weighing 0.0001 g
Bottle top dispenser	Suitable for procedure
Centrifuge	Eppendorf Centrifuge 5810
High-speed plate shaker	SPEX Geno/Grinder® 2010 or comparable
<b>Sample Preparation Consumables</b>	
Filter Paper	Whatman™ Qualitative Filter Papers 3, 125mm diameter, Cat. No. 1003-125
Grinding ball, 7/16 inch	OPS Diagnostics Cat. No. GBSS 437-1003-03
Polypropylene extraction tubes (50 mL)	SARSTEDT Cat. No. 62.548.304
2 mL glass sample vials with cap and PTFE/SIL septa	Xpertek /PJ Cobert Cat. No. 958933

Monsanto Company Method

Effective Date: April 20, 2018 ME-2166-01 Page 2 of 9  
Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

**Chemicals & Reagents** The following chemicals and reagents are used in this method. Specific brands are listed to aid the analyst in finding items. In most cases, equivalent chemicals and reagents from other vendors may be used.

It is important to use high quality reagents to avoid chromatographic interferences.

Chemical/Reagent	Number/Specification
ACN, HPLC grade	Burdick & Jackson Cat. No. 015-4
Water, HPLC grade	Millipore Water System
Dicamba	Monsanto ARS Program or commercially available
Phosphoric Acid, 85% ( $H_3PO_4$ )	Fisher Cat # A260-500
50% Sodium Hydroxide	J.T. Baker #5666-02

(Refer to [Appendix A](#) for analyte standard compound structures.)

### Reagent/Solution Preparation

Prepare the following reagent solutions for use in sample analysis. The absolute volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained. A six-month expiration date will be assigned to these solutions unless a shorter expiration is specified on the reagent container label. Solutions may be stored at room temperature in glass containers, unless otherwise specified.

Solution	Preparation
0.1M Phosphoric Acid in Water (HPLC Mobile Phase A)	4000 mL of water mixed with 45.8 g 85% $H_3PO_4$ .
ACN (HPLC Mobile Phase B)	N/A
12.5 mM NaOH	1 g 50% NaOH/ 1000 mL HPLC grade water.

### Standard Calibration and QC Solution Preparation

**Overview** All standard solutions must be properly labeled and stored in glass bottles with airtight lids in a refrigerator (i.e. approximately 4°C). Preparation procedures which result in equivalent solutions may be substituted. Various additional solutions may be prepared as long as the preparation is documented.

**Stability** The recommended expiration date for the dicamba acid standard solutions when stored in a refrigerator is six months.

**Preparation and Calculations for Standard Stock Solutions** Solutions may be prepared in the following manner. Other concentrations may be used as long as the preparation is documented. A suggested scheme for stock and working calibration solution preparation is shown below. Additional solution levels may be prepared as necessary. 12.5 mM NaOH is suggested for use as the diluent for the stock solutions as it aids in the dissolution of dicamba acid. Water can be substituted if care is taken to assure dissolution by mixing.

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 3 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

**Stock Solution 1 (approximately 0.2% by weight):**

Weigh approximately 0.1 grams dicamba acid reference standard to the nearest 0.0001 gram into a tared bottle. Add approximately 50 ml 12.5 mM NaOH and weigh the total (standard +water) to the nearest 0.0001 gram. Mix until solubilized.

**Stock Solution 2 (approximately 0.05% by weight):**

Weigh approximately 0.025 grams dicamba acid reference standard to the nearest 0.0001 gram into a tared bottle. Add approximately 50 ml 12.5 mM NaOH and weigh the total (standard +water) to the nearest 0.0001 gram. Mix until solubilized. Calculate actual standard concentrations based on the actual weights used and the purity of the reference standard.

**Working  
Calibration  
Standard  
Solutions**

Solutions may be prepared in the following manner. Aliquots of stock solutions are diluted with water and mixed. Other concentrations may be used as long as the preparation is documented. A suggested scheme for working calibration solution preparation is shown below. Additional solution levels may be prepared as necessary. Calculate actual standard concentrations based on the calculated concentrations of the stock solutions and the actual weights taken.

**Dicamba Working Calibration Solutions**

Working Calibration Solution (wt%)	Aliquot Solution Concn (wt%)	Aliquot Weight (g)	Water Weight (g)
0.001	0.05	0.2	9.8
0.003	0.05	0.6	9.4
0.005	0.2	0.25	9.75
0.01	0.2	0.5	9.5
0.02	0.2	1	9.0

**QC  
Fortification  
Solutions**

QC solutions are prepared from a source formulation that can represent the formulation(s) used within a study. For example, Xtendimax should be used as a QC for dicamba formulations and tank mixes and Roundup Xtend should be used as a QC for dicamba/glyphosate formulations and tank mixes. The final concentration of the QC fortification solutions is based on study parameters (i.e. the concentration of the solution that is to be sprayed on the filter paper). An example scheme for QC fortification solution is shown below.

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 4 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

Example of Dicamba QC Fortification Solutions Preparation

Source Formulation	Source Conc (wt% dicamba acid)	Source Aliquot weight (g)	Diluent weight (g)	QC Spike Solution Conc (wt% dicamba)	mg Dicamba per filter paper with a 0.3 g spike <sup>1</sup>
XtendiMax (MON 76980)	29.0	0.200	11.25	0.50 wt%	1.5
Roundup Xtend (MON 76981)	9.8	0.5	9.3	0.50 wt%	1.5

<sup>1</sup>This is the expected amount of dicamba on each QC filter paper after application of 0.3 grams of the QC fortification solution (step 2 of sample processing).

Sample Preparation Procedure

**Sample Storage** Samples will be maintained frozen at approximately -20 °C for extended storage periods.

**Sample Processing** The following describes the preparation of samples for dicamba analysis by HPLC-UV. A typical analytical set will include study samples, QCs and standards.

Step	Action
1	Add a single piece of filter paper to each tube designated as a QC.
2	For each test substance, add the target weight of the appropriate QC spiking solution for each level of QC as indicated <sup>1</sup> . Record each weight in g to four decimal places:  For example, if the target weight of dicamba on the QC filter paper is 1.5 mg add 0.3 g of a 0.50 wt% QC Fortification Solution.
3	Place each filter paper sample individually in a 50 ml extraction tube.
4	To all samples and QCs add ~30 g of water and record each weight in g to 4 decimal places.
5	Add one grinding ball to each tube.
6	Place a cap on each tube. Ensure the cap is sealed well before proceeding.
7	Shake samples on the Geno/Grinder® to extract analyte from the filter paper (e.g., 1200 cycles per minute for 5 minutes). Examine the tubes for leaks. If leaks are detected, discard and re-prepare.
8	Centrifuge tubes to pellet paper (e.g. 5 minutes at 4500 x g) and filter supernatant through a PTFE filter (0.45 µm recommended) before transferring to 2 ml autosampler vials.
9	Analyze the solution by HPLC-UV.
Note: <sup>1</sup> These spike solution target weights may be changed based on the study parameters (expected amount of dicamba on filter papers).	

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 5 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

**Instrumental Analysis**

<b>Sample Analysis Guidance and Acceptance Criteria</b>	<p>The requirements for sample analyses include but are not limited to the following:</p> <ul style="list-style-type: none"> <li>• An analytical set contains calibration standards, QC fortifications and test samples.</li> <li>• Analyte calibration must be performed for each set using a calibration curve with a minimum of five calibration levels (excluding blanks). The curve should span at least from 60-140% of the target value of the expected analyte concentration of the prepared samples. The coefficient of determination (<math>R^2</math>) must be <math>\geq 0.999</math>.</li> <li>• Responses for the highest and lowest standards must bracket those of the study samples.</li> <li>• Each batch run must contain a minimum of triplicate QC fortifications at two levels. QC fortifications must have a mean accuracy range of 90-110% at each QC level and a precision of <math>\leq 5\%</math> RSD at each QC level.</li> <li>• The calculated concentration of the study samples is expected to be within 90-110% of the expected analyte concentration in the study sample(s). If the calculated concentration is outside of this range the study director will be notified and at the study director's discretion additional testing (such as standard addition or diode array detection) may be utilized to determine if interferants are present.</li> <li>• Acceptance of data that do not meet the above criteria must have a documented reason and approval by the Study Director.</li> </ul>
<b>Instrument Setup</b>	<p>The recommended method parameters are those that have been described below. However, the mobile phase, HPLC column type and temperature, flow rate, injection size, and detector setting may be changed as long as acceptable chromatographic performance such as calibration, linearity, and chromatographic separation are achieved.</p>

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 6 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

**System  
Conditions for  
Analysis**

**HPLC-UV System Conditions**

Column: HiChrom Alltima C18, 250 mm × 4.6 mm, 5 µm  
Injection volume: 20 µL  
Column oven temperature: 30 °C  
Detector Wavelength: 280 nm  
Mobile Phase A: 0.1 M Phosphoric Acid in Water  
Mobile Phase B: Acetonitrile  
Flow Rate: 1.5 mL/min  
HPLC Gradient Conditions:

Time (min)	%B
0	40
1	40
10	100
15	100
16	40
20	40

Total Run time: 20 min

**Calculations**

**Calculations for  
Standard  
Solutions and  
QC  
Fortifications**

The concentration of the standard stock solutions in weight percent units is calculated by the following equation:

$$\text{Wt \% dicamba} = \frac{(\text{grams dicamba}) \times (\text{purity}) \times 100}{(\text{grams total solution wt})}$$

The weight percent concentration of each working solution and QC fortification solution is calculated as follows:

$$\text{Wt \%} = \frac{(\text{grams stock solution})}{(\text{grams total solution})} \times (\text{Wt \% dicamba in stock solution})$$

**Calculations for  
Analyte result**

The method uses an external standard with 3 or more standard levels for dicamba calibration. Linear regression analysis is used to construct a line with the peak area responses of the dicamba for the standards (on the y-axis) versus the concentration of dicamba in the standards (on the x-axis).

This equation is of the form:

$$Y = MX + B$$

The equation for the regression line is then converted to a prediction equation by solving for x as a function of y:

$$X = (Y - B) / M$$

Where X is the concentration of dicamba in units of wt%,

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 7 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

Y is the area of dicamba,  
M is the slope as determined from the regression equation, and  
B is the y-intercept as determined from the regression equation.

The concentration of dicamba in the prepared sample is found using the equation as follows:

$$\text{Dil Wt\%} = \frac{(\text{Sample Area dicamba} - B)}{M}$$

Dil Wt% is the dicamba acid weight percent in the diluted sample.

Analyte concentrations are reported as mg dicamba acid/filter paper. The amount of dicamba on each filter paper is found using the equation as follows:

$$\text{mg dicamba acid/filter paper} = \text{Dil Wt\%} * \text{wt diluent added (g)} * 10$$

**Calculations for Recovery of QC samples**

Recovery of dicamba from the QC samples is calculated by comparing the amount of dicamba found in the samples to the calculated amount of dicamba that was spiked.

To calculate the amount of dicamba spiked per filter paper:

$$\text{Amount spiked (mg)} = \text{Wt spiking solution (g)} * \text{Conc of spike solution (wt\%)} * 10$$

To calculate the amount of dicamba found by assay see the preceding section "Calculations for Analyte Result."

The recovery is calculated

$$\% \text{ Recovery} = \frac{\text{Amount dicamba found by assay (mg)} * 100}{\text{Amount dicamba spiked (mg)}}$$

Monsanto Company Method

Effective Date: April 20, 2018

ME-2166-01

Page 8 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

Documentation

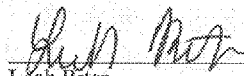
The analytical raw data packages will include (at a minimum): record of sample weights/dispensing, sample processing worksheets, instrumental sample queue/run record, calibration curves, MRM chromatograms, results tables, and instrument acquisition parameters.

**Note:** Method validation data and example chromatograms for calibration standards, control samples, and fortified samples are documented in MSL0029468.

Author(s): Leah Riter

Approval

Author:



Leah Riter

(Analytical Methods and Analysis Lead, Monsanto Company)

Date Apr 18 / 2018

Management:



Michael R. Shepard

(Struct Elucidation & Residue Analysis Lead, Monsanto Company)

Date Apr 18 / 2018



Monsanto Company Method

Effective Date: April 20, 2018

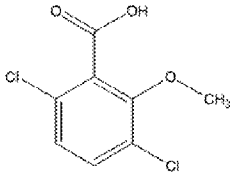
ME-2166-01

Page 9 of 9

Determination of Dicamba on Filter Paper by HPLC-UV for Application Rate Verification

Appendices

Appendix A: Chemical Structures

	<p>Dicamba</p> <p>3,6-dichloro-2-methoxybenzoic acid</p> <p>CAS # 1918-00-9</p> <p><math>C_8H_6Cl_2O_3</math></p> <p>Average Molecular weight: 221.04</p>
---	---

## Appendix C. Analytical Method – Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

Monsanto Company Method

Effective Date: April 20, 2018

ME-2154-01

Page 1 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

### Overview

**Purpose & Scope** This document describes the method used by used by Environmental Sciences personnel to determine dicamba acid concentration in dicamba tank mixes used in field applications.

**Method Summary** Dicamba tank mix samples are brought to room temperature, then gently shaken to appropriate homogeneity to allow reproducible measurements of 0.5 grams. A sufficient amount of DI water is used to dilute the sample to a final concentration of approximately 0.1% dicamba. The DI water is weighed into a bottle followed by addition of 0.5 grams of the tank mix sample. The samples are capped and gently shaken. An aliquot is transferred to an autosampler vial for quantitation using HPLC-UV. The working range of the method is from 0.06 to 0.14% (wt/wt) dicamba.

**Safety Precautions** Observe the usual safety procedures in handling laboratory equipment, chemicals, and solvents in accordance with local safety regulations. Read and follow all safety precautions on the labels of all chemicals used in this procedure.

### Materials

**Equipment** The following equipment is used in this procedure. Specific brands are listed to aid the analyst in finding items. In most cases, equivalent equipment from other vendors can be used.

Equipment	Number/Specification
HPLC system	Gradient pump capable of pumping at 1.5 mL per minute, such as a Varian ProStar HPLC pump, a UV detector, such as a Varian ProStar UV detector, and an autosampler capable of accurately and precisely injecting a defined volume, such as a Varian ProStar autosampler.
HPLC column	HiChrom Alltima C18, 250 x 4.6 mm, 5µm
Analytical balance	Capable of weighing 0.0001 g
4 oz. glass bottles with tight fitting screw cap.	Suitable for procedure
2.0 mL autosampler vials.	Suitable for procedure
Glass pipettes	Suitable for procedure

**Chemicals & Reagents** Specific brands are listed to aid the analyst in finding items. Generally, equivalent reagents and standards obtained from other vendors may be substituted for the specified product.

Chemical/Reagent	Catalog Number
Acetonitrile, HPLC or higher purity grade	Burdick & Jackson Cat. No. 015-4
Water, HPLC or higher purity grade	J.T. Baker Cat. No. 4218-03
Phosphoric Acid, 85%	Fisher, Cat. No. A260-500
50% Sodium Hydroxide	J.T. Baker, Cat. No. 5666-02

Monsanto Company Method

Effective Date: April 20, 2018 ME-2154-01 Page 2 of 7  
Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

Dicamba	Monsanto ARS program or commercially available
---------	--

(Refer to [Appendix A](#) for analyte standard compound structures.)

**Reagent/Solution Preparation**

Prepare the following reagent solutions for use in sample analysis. The absolute volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained. A six-month expiration date will be assigned to these solutions unless a shorter expiration is specified on the reagent container label. Solutions may be stored at room temperature in glass containers, unless otherwise specified.

Solution	Preparation
0.1M Phosphoric Acid in Water (HPLC Mobile Phase A)	4000 mL of water mixed with 45.8 g 85% $H_3PO_4$ .
ACN (HPLC Mobile Phase B)	N/A
12.5 mM NaOH	1 g 50% NaOH/ 1000 mL HPLC grade water.

**Standard Solution Preparation**

<b>Overview</b>	All standard solutions must be properly labeled and stored in glass bottles with airtight lids in a refrigerator (i.e. approximately 4°C). Preparation procedures which result in equivalent solutions may be substituted. Various additional solutions may be prepared as long as the preparation is documented.
<b>Stability</b>	The recommended expiration date for the dicamba acid standard solutions when stored in a refrigerator is six months.

Monsanto Company Method

Effective Date: April 20, 2018

ME-2154-01

Page 3 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

**Dicamba Calibration Solutions** Prepare standard solutions to cover the required calibration range. A minimum of 5 standards is recommended. The following is a guideline for a 5-point calibration standard preparation. Other concentrations and amounts may be prepared.  
**Note:** purity of the analytical reference standard must be used to calculate final % dicamba in calibration standards.

Standard level	Dicamba(g)	% Dicamba
Std. 1	0.0300	0.0600
Std. 2	0.0400	0.0800
Std. 3	0.0500	0.1000
Std. 4	0.0600	0.1200
Std. 5	0.0700	0.1400

- Weigh amounts of dicamba acid standard according to the guideline above into clean dry 4 oz glass bottles. Record weights to the nearest 0.0001 g.
- Add 50 g of 12.5 mM NaOH solution. Record weights to the nearest 0.0001 g. Sonicate for at least 15 minutes until no solid particle is visible.

**QC Fortification Solutions**

QC solutions are prepared from a source formulation that can represent the formulation(s) used within a study. For example, Xtendimax should be used as a QC for dicamba formulations and tank mixes and Roundup Xtend should be used as a QC for dicamba/glyphosate formulations and tank mixes. The final concentration of the QC fortification solutions is based on study parameters (i.e. the concentration of the solution that is to be sprayed in the field). An example scheme for QC fortification solution is shown below.

Example of Dicamba QC Fortification Solutions Preparation

Source Formulation	Source Conc (wt% dicamba acid)	Source Aliquot weight (g)	Diluent weight (g)	QC Spike Solution Conc (wt% dicamba)
XtendiMax (MON 76980)	29.0	0.200	11.25	0.50 wt%
Roundup Xtend (MON 76981)	9.8	0.5	9.3	0.50 wt%

**Sample Preparation Procedure**

**Sample Storage** Samples will be maintained refrigerated at approximately 4 °C when not in use.

**Sample Homogenization** Tank mix samples must be homogenized prior to analysis by gentle shaking by hand to produce representative 0.5 g subsamples.

Monsanto Company Method

Effective Date: April 20, 2018

ME-2154-01

Page 4 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

Sample Processing

The following describes the preparation of samples for dicamba analysis.

Step	Action
1	<p>Weigh a sufficient amount of DI water into a glass bottle to dilute the sample to a final target concentration of approximately 0.1% (wt/wt) dicamba. Record weights to the nearest 0.0001 g</p> $DI\ H_2O\ (grams) = \left( \frac{Nom.\ Dicamba\ (\%wt/wt) \times 0.5\ g\ aliquot}{0.1\% \ (target\ dicamba\ load)} \right) - 0.5\ g$ <p>For example: Nominal Dicamba % = 1.2% (wt./wt.)</p> $DI\ H_2O\ (grams) = \left( \frac{1.2\% \times 0.5\ g}{0.1\%} \right) - 0.5\ g$ <p>Add 5.5 grams of water.</p>
2	<p>After bringing samples to room temperature, weigh approximately 0.5 g of sample into the glass bottle. Record weights to the nearest 0.0001 g. Cap the sample and shake gently until well mixed. <b>Note that the order of addition, water then sample, is important.</b> Do not use plastic bottles.</p>
3	<p>Transfer an aliquot using a glass pipette to autosampler vials for analysis.</p>
4	<p>Analyze by HPLC-UV within storage time determined during method validation.</p>

Instrumental Analysis

Sample Analysis

The requirements for sample analyses include but are not limited to the following:

Guidance and Acceptance Criteria

- An analytical set contains calibration standards, QC fortifications and test samples.
- Analyte calibration must be performed for each set using a calibration curve with a minimum of five calibration levels (excluding blanks). The curve should span at least from 60-140% of the target value of the expected analyte concentration of the prepared samples. The coefficient of determination ( $R^2$ ) must be  $\geq 0.999$ .
- Responses for the highest and lowest standards must bracket those of the study samples.
- Each batch run must contain a minimum of triplicate QC fortifications at two levels. QC fortifications must have a mean accuracy range of 90-110% at each QC level and a precision of  $\leq 5\%$  RSD at each QC level.
- The calculated concentration of the study samples is expected to be within 90-110% of the expected analyte concentration in the study sample(s). If the calculated concentration is outside of this range the study director will be notified and at the study director's discretion additional testing (such as standard addition or diode array detection) may be utilized to determine if interferants are present.
- Acceptance of data that do not meet the above criteria must have a documented reason and approval by the Study Director.

Monsanto Company Method

Effective Date: April 20, 2018

ME-2154-01

Page 5 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

**Instrument Setup** Instrument operation is controlled by acquisition methods containing all autosampler, HPLC, and UV detector operating parameters. The following equipment and conditions is instrument dependent and may be modified to obtain optimal instrument performance and maximize sensitivity. The mobile phase, HPLC column, flow rate, injection size, and detector settings may be changed if acceptable chromatographic performance, such as calibration linearity and chromatographic separation, are achieved. Actual method parameters must be documented in the raw data.

HPLC-UV System Conditions

Column: HiChrom Alltima C18, 250 x 4.6 mm, 5µm or equivalent

Pump flow rate: 1.5 mL/minute

HPLC column temperature: 30 °C

Injection volume: 20µL

UV detection wavelength: 280nm

Mobile Phase A: 0.1 M Phosphoric Acid in Water

Mobile Phase B: ACN

HPLC Gradient Conditions:

Time (min)	%B
0.0	40
1	40
10	100
15	100
16	40
20	40

Typical retention time for dicamba: 7 minutes

Monsanto Company Method

Effective Date: April 20, 2018

ME-2154-01

Page 6 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

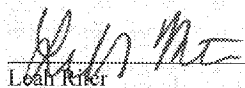
Documentation

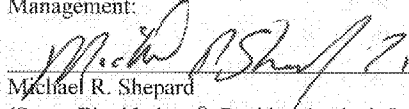
The analytical raw data packages will include (at a minimum): record of sample weights/dispensing, sample processing worksheets, instrumental sample queue/run record, calibration curves, MRM chromatograms, results tables, and instrument acquisition parameters.

**Note:** Method validation data and example chromatograms for calibration standards, control samples, and fortified samples are documented in MSL0029467.

Author(s): Leah Riter

Approval

Author:  
  
Leah Riter  
(Analytical Methods and Analysis Lead, Monsanto Company)  
Date Apr / 18 / 2018

Management:  
  
Michael R. Shepard  
(Struct Elucidation & Residue Analysis Lead, Monsanto Company)  
Date Apr / 18 / 2018

Monsanto Company Method

Effective Date: April 20, 2018

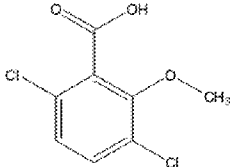
ME-2154-01

Page 7 of 7

Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification

Appendices

Appendix A: Chemical Structures

	<p>Dicamba</p> <p>3,6-dichloro-2-methoxybenzoic acid</p> <p>CAS # 1918-00-9</p> <p><math>C_8H_6Cl_2O_3</math></p> <p>Average Molecular weight: 221.04</p>
---	---



ABC Laboratories, Inc.

CD001 (10APR14)

**Observations and/or Remarks**

Study No.: 89311

Page 1 of 1

**Eurofins Laboratories Method Modifications to Monsanto Method ME-2154-01**

*Determination of Dicamba Acid in Tank Mixes by HPLC-UV for Application Rate Verification*

**Reason for Modifications:**

- 1) To document alternate preparation for calibration standards routinely used in sample analysis. This alternate preparation is used so that final composition of calibration standards more closely represents the final composition of tank mix samples diluted for analysis.

**Working Calibration Standard Solutions**

Solutions may be prepared in the following manner. Aliquots of stock solutions in 12.5 mM NaOH are diluted with water and mixed. Other concentrations may be used as long as the preparation is documented. A suggested scheme for working calibration solution preparation is shown below. Additional solution levels may be prepared as necessary. Calculate actual standard concentrations based on the calculated concentrations of the stock solutions and the actual weights taken.

**Dicamba Working Calibration Solutions**

Working Calibration Solution (µg/g)	Aliquot Solution Concn (mg/g)	Aliquot Weight (g)	Water Weight (g)
250	1.0	12.5	37.5
100	1.0	5.0	45.0
50	1.0	2.5	47.5
25	1.0	1.25	48.75
10	1.0	0.50	49.5
5.0	1.0	0.25	49.75

Prepared by: MRE 956A9

## Appendix D. Analytical Method – LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 1 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

### Overview

**Purpose & Scope** This procedure describes an analytical method used for the determination of dicamba on Polyurethane Foam (PUF) air sampling traps.

**Method Summary** Dicamba is extracted from polyurethane foam (PUF) air sampling traps using methanol containing stable-labeled internal standard. The sample tubes are capped and agitated on a high-speed shaker for extraction. A 10-mL aliquot of the supernatant is filtered, evaporated to approximately 10% of its original volume, then re-filtered and evaporated to dryness. The samples are reconstituted in 0.10 mL of 25% methanol in water, yielding a 100-fold concentration factor. Dicamba is then quantitated using LC-MS/MS with electrospray ionization in negative ion mode with SelexION<sup>®</sup>+ Differential Mobility Separation. The working range of the method is from 0.03 to 7.5 ng/PUF (0.10 to 25.0 ng/mL injected on column), with an LOQ of 0.1 ng/PUF. Extension of the upper range of the method using lower injection volumes was demonstrated during method validation.

**Safety Precautions** Follow appropriate safety policies. Important precautions include:

- Some solvents are volatile and/or flammable. Care must be taken to keep them away from any source of ignition.
- Ensure proper ventilation to avoid excessive exposure to toxic solvent vapors.
- Read and follow all safety warnings on reagent containers.

**Abbreviations** The following abbreviations are used in this method:

Abbreviation	Definition
ACS	American Chemical Society
ACN	acetonitrile
amu	atomic mass unit
Approx.	approximately
ARS	Analytical Reference Standard
CAD	collision assisted dissociation
CE	collision energy
Concn	concentration
CUR	curtain gas
CXP	collision cell exit potential
DP	declustering potential
EP	entrance potential
ESI	electrospray ionization
g	gram
HPLC	high-performance liquid chromatography
IS	internal standard
L	liter
LC-MS/MS	liquid chromatography tandem mass spectrometry
LOD	limit of detection
LOQ	limit of quantitation

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 2 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

MeOH	methanol
min	minute
mL	milliliter
mm	millimeter
MRM	multiple reaction monitoring
ms	millisecond
MS	mass spectrometry
N	number of samples
N/A	not applicable
ND	not detected
N <sub>2</sub>	nitrogen
ng	nanogram
PTFE	polytetrafluoroethylene
PUF	polyurethane foam
Q	quadrupole
QC	quality control
RSD	relative standard deviation
RT	room temperature
µg	microgram
µl	microliter
µm	micrometer
V	volt

Contents

Section	Page
Overview	1
Materials	3
Reagent/Solution Preparation	4
Standard Calibration and QC Solution Preparation	5
Sample Preparation Procedure	9
Instrumental Analysis	11
Calculations	13
Documentation	14
<b>Appendices</b>	
Appendix A: Compound Structures	16
Appendix B: Representative Chromatograms	17
Appendix C: Validation Summary	18

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 3 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Materials**

**Equipment**

The following equipment/supplies are used in this procedure. Specific brands are listed to aid the analyst in finding items. In most cases, equivalent equipment from other vendors can be used.

Equipment	Number/Specification
<b>Analytical Instruments</b>	
Mass spectrometer	AB Sciex API 6500™+ with Turbo-V ionization source and SelexION®+
Data acquisition system	PC workstation with AB Sciex Analyst® software
HPLC system	AB Sciex ExionLC™ AD System: Solvent Degasser, at least 2 Pumps, Autosampler, Heated Column Compartment and Controller
HPLC switching valve	Rheodyne, 6 port
HPLC column	Phenomenex Kinetex Biphenyl 50 mm × 3.0 mm, 2.6 µm
<b>Sample Preparation Equipment</b>	
Analytical balance	Capable of weighing 0.0001 g
Bottle top dispenser	BrandTech Scientific, Cat. No. 4731361
Graduated cylinder (100 mL, 1 L)	Suitable for procedure
High-speed plate shaker	SPEX Geno/Grinder® 2010 or comparable
Mechanical pipettes	suitable for procedure
Bottle top dispenser	BrandTech® Dispensette® Organic
Sample concentrator	SPE Dry 96, Biotage
Nitrogen evaporator	Organomation N-Evap
<b>Sample Preparation Consumables</b>	
Polyurethane Foam (PUF), 22 mm diameter × 76 mm length	SKC Inc. Cat. No. P22692
Grinding ball, 1/4 inch	Midway USA, 1/4" stainless steel shot, Part No. SH1F
Polypropylene extraction tubes (50 mL)	SARSTEDT Cat. No. 62.548.304
Polypropylene extraction tubes (15 mL)	Fisher Cat. No. 14-959-70C
96-Well filter plate, 0.45 µm polypropylene (2 mL), long drip	Agilent PN 201009-100
12 mL amber glass sample vials with PTFE lined cap	Fisher Cat. No. 03-391-8D
16 mL amber glass sample vials with PTFE lined cap	Fisher Cat. No. 03-391-8E
96-Well square well sealing mat	Analytical Sales & Services, Inc. Cat. No. 964085
96 Deep-well plate, square, tapered well polypropylene (2 mL)	Analytical Sales & Services, Inc. Cat. No. 968820

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 4 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Chemicals & Reagents**

The following reagents are used in this method. Specific brands are listed to aid the analyst in finding items. Generally, equivalent reagents and standards from other vendors may be substituted for the specified product. It is important to use high quality reagents to avoid chromatographic interferences. It is recommended to verify the isotopic purities of the internal standard materials prior to use.

Chemical/Reagent	Number/Specification
ACN, HPLC grade	Burdick & Jackson Cat. No. 015-4
Water, HPLC grade	J.T. Baker Cat. No. 4218-03
Methanol, HPLC grade	EMD Cat. No. MX0488-1
Ethanol, ACS grade	Sigma Cat. No. 459844-1L
2-propanol, HPLC grade	Fisher Cat No. A451-4
Formic acid, LC-MS grade	Sigma Cat. No. 56302-50ML
Dicamba	Monsanto ARS Program or Commercially available (e.g. Sigma Cat. No. 16826)
( <sup>13</sup> C <sub>6</sub> )Dicamba	Monsanto ARS Program or Commercially available (e.g. Sigma Cat. No. 705306)

(Refer to [Appendix A](#) for analyte and internal standard compound structures.)

**Reagent/Solution Preparation**

Prepare the following reagent solutions for use in sample analysis. The absolute volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained. A six-month expiration date will be assigned to these solutions unless a shorter expiration is specified on the label. Solutions may be stored at room temperature in glass containers, unless otherwise specified.

Solution	Preparation
HPLC Mobile Phase A	0.05% formic acid in water: Add 0.5 mL formic acid to 1000 mL water
HPLC Injection Needle Wash	25% MeOH in water: Add 750 mL water to 250 mL of methanol
25% MeOH in water	Add 750 mL water to 250 mL of methanol

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 5 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Standard Calibration and QC Solution Preparation**

**Overview** All stock, fortification, and calibration solutions must be properly labeled and stored in amber glass vials with airtight lids at approximately 4 °C. Preparation procedures which result in equivalent solutions may be substituted, including directly weighing or diluting standards in amber glass bottles by known diluent addition (to the nearest 0.1 mL). Various additional solutions may be prepared as long as the preparation is documented. Stock solutions must be adjusted for purity (purity adjustment is not needed for internal standards).

**Stability** The stability of dicamba in ethanol was demonstrated during the validation of method ME-1321. The stability of dicamba in ACN was demonstrated during the validation of method ME-1381.

Solution Components	Solution Type	Concentration or Range <sup>1,2</sup>	Solvent	Approx. Storage (°C)	Demonstrated Stability (Days)
Dicamba	Stock Solution	100 µg/mL <sup>3</sup>	Ethanol	4	201
Dicamba	Intermediate and Working Calibration Standard Solutions	0.010 to 25 µg/mL	ACN	4	201

<sup>1</sup> Stability of the stock solutions was established in method ME-1321.

<sup>2</sup> Stability of intermediate and working solutions was established in method ME-1381.

<sup>3</sup> According to SOP PR-0897 ("Quantitative Analytical Reference Standard Solution Stability"), it is scientifically reasonable to assume that concentrations above the highest evaluated standard level would also be stable during the same length of storage (except in cases where solubility might be a concern). Therefore, in this method, the stability of dicamba in ethanol is extended to the 0.50 mg/mL QC Stock Solution.

**Dicamba Calibration Stock Solution (0.50 mg/mL)** Weigh 5-10 mg (recorded to at least 0.1 mg) of dicamba standard into an appropriate volumetric flask (or appropriate weigh boat and quantitatively transfer to the flask using ethanol). Adjust the volume appropriately (bring to volume in a volumetric flask) using ethanol to prepare a 0.50 mg/mL solution (purity adjusted). The solution should be mixed or vortexed until completely dissolved. The use of a sonicator may be used to facilitate complete dissolution if needed. Transfer the solution to an amber glass bottle for storage. Alternatively, weigh 5-10 mg (recorded to at least 0.1 mg) of standard into an amber glass bottle. Add the appropriate volume (to the nearest 0.1 mL) of ethanol to prepare a 0.50 mg/mL solution (purity adjusted). An adjustable positive-displacement mechanical pipette capable of delivering up to 50 mL is recommended. The solution should be mixed or vortexed until completely dissolved. The use of a sonicator may be used to facilitate complete dissolution if needed.

Note: The absolute mass and volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained.

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 6 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Dicamba QC  
Stock Solution  
(0.50 mg/mL)**

Prepare a separate 0.5 mg/mL (purity adjusted) Dicamba QC Stock Solutions Stock Solution using the procedure for the Dicamba Calibration Stock Solution.

Note: The absolute mass and volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained.

**Intermediate  
Calibration  
Solutions**

Prepare the following intermediate standards by dilution of the appropriate stock solution in amber glass vials with ACN. These solutions will be used for the preparation of working calibration standard solutions.

Intermediate Calibration Solution (µg/mL)	Aliquot Solution ID	Aliquot Volume (mL)	Diluent Volume (mL)
10	Dicamba Calibration Stock Solution (0.50 mg/mL)	0.50	24.5
1.0	10 µg/mL Intermediate Calibration Solution	1.00	9.00
0.10	1.0 µg/mL Intermediate Calibration Solution	1.00	9.00
0.010	0.10 µg/mL Intermediate Calibration Solution	1.00	9.00

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 7 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

**Working  
Calibration  
Standard  
Solutions**

A suggested scheme for working calibration solution preparation is shown below. Solutions may be prepared in the following manner. Other concentrations may be used as long as the preparation is documented. For each solution, add the listed aliquot of the designated solution to an amber glass vial and dilute with the specified volume of ACN. Additional solution levels may be prepared as necessary.

**Dicamba Working Calibration Solutions**

Working Calibration Solution (µg/mL)	Aliquot Solution Conc (µg/mL)	Aliquot Volume (mL)	Diluent Volume (mL)	Equivalent Conc <sup>1</sup> (ng/PUF)	Concn at Injection <sup>2</sup> (ng/mL)
0.00030	0.010	0.30	9.70	0.030	0.10
0.00075	0.010	0.75	9.25	0.075	0.25
0.0010	0.10	0.10	9.90	0.10	0.33
0.0015	0.10	0.15	9.85	0.15	0.50
0.0030	0.10	0.30	9.70	0.30	1.0
0.0075	0.10	0.75	9.25	0.75	2.5
0.010	1.0	0.10	9.90	1.0	3.33
0.015	1.0	0.15	9.85	1.5	5.0
0.030	1.0	0.30	9.70	3.0	10
0.075	1.0	0.75	9.25	7.5	25

<sup>1</sup> This concentration represents 0.100 mL of Working Calibration Solution diluted by the extraction solvent (30 mL). For example, 0.100 mL of the 0.015 µg/mL Working Calibration Solution diluted to 30 mL results in an equivalent concentration of 1.5 ng dicamba/PUF.

<sup>2</sup> This concentration represents 0.100 mL of Working Calibration Solution diluted by the extraction solvent (30 mL), then concentrated 100-fold during sample processing. For example, 0.100 mL of the 0.015 µg/mL Working Calibration Solution diluted to 30 mL, a 10 mL aliquot is dried and reconstituted in 0.10 mL results in an equivalent concentration of 0.50 ng dicamba/mL injected on column.



Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 8 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

**QC  
Fortification  
Solutions**

Solutions may be prepared in the following manner. Other concentrations may be used as long as the preparation is documented. A suggested scheme for QC fortification solution preparation is shown below. For each fortification solution, add the listed aliquot of the designated solution to an amber glass vial and dilute with the specified volume of ACN. Additional fortification solution levels may be prepared as necessary.

**Dicamba QC Fortification Solutions**

QC Solution Concn ( $\mu\text{g/mL}$ )	Aliquot Solution ID	Aliquot Volume (mL)	Diluent Volume (mL)	Fortification Concn (ng/PUF) <sup>1</sup>
5.0	Dicamba QC Stock Solution 0.50 mg/mL Dicamba	0.25	24.75	NA
0.10	5.0 $\mu\text{g/mL}$ Dicamba QC Solution	0.20	9.80	NA
0.0010	0.10 $\mu\text{g/mL}$ Dicamba QC Solution	0.10	9.90	0.10 (LOQ QC)
0.010	0.10 $\mu\text{g/mL}$ Dicamba QC Solution	1.0	9.0	1.0 (Mid QC)
0.060	5.0 $\mu\text{g/mL}$ Dicamba QC Solution	0.12	9.88	6.0 (High QC)
0.60	5.0 $\mu\text{g/mL}$ Dicamba QC Solution	1.2	8.8	60 (Dilution QC)

<sup>1</sup> Concentration represents 0.100 mL of QC Fortification Solution diluted by the extraction solvent. For example, 0.100 mL of the 0.010  $\mu\text{g/mL}$  QC Solution diluted to 30 mL resulted in an equivalent concentration of 1.0 ng dicamba/PUF.

**(<sup>13</sup>C<sub>6</sub>)Dicamba  
IS Stock  
Solution  
(0.10 mg/mL)**

Weigh 4-5 mg (recorded to at least 0.1 mg) of (<sup>13</sup>C<sub>6</sub>) Dicamba standard into an appropriate volumetric flask (or appropriate weigh boat and quantitatively transfer to the flask using ethanol). Adjust the volume appropriately (bring to volume in a volumetric flask) using ethanol to prepare a 0.10 mg/mL solution (adjustment for purity is not necessary). The solution should be mixed or vortexed until completely dissolved. The use of a sonicator may be used to facilitate complete dissolution if needed. Transfer the solution to an amber glass bottle for storage. Alternatively, weigh 4-5 mg (recorded to at least 0.1 mg) of standard into an amber glass bottle. Add the appropriate volume (to the nearest 0.1 mL) of ethanol to prepare a 0.10 mg/mL solution (do not adjust for purity). An adjustable positive-displacement mechanical pipette capable of delivering up to 50 mL is recommended. The solution should be mixed or vortexed until completely dissolved. The use of a sonicator may be used to facilitate complete dissolution if needed.

Note: The absolute mass and volume of the solutions may be varied at the discretion of the analyst, as long as the correct proportions of the components are maintained.

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 9 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**IS Working Solution (0.03 µg/mL)** Solutions may be prepared in the following manner. A suggested scheme for IS solution preparation is shown below. For each IS solution, add the listed aliquot of the designated solution to an amber glass vial and dilute with the specified volume of ACN.

IS Solution Conc'n (µg/mL)	Aliquot Solution ID	Aliquot Volume (mL)	ACN Volume (mL)
10	( <sup>13</sup> C <sub>6</sub> )Dicamba IS Stock Solution (0.10 mg/mL)	1.0	9.0
0.030	10 µg/mL Dicamba IS Solution	0.3	99.7

**Sample Preparation Procedure**

**Sample Storage** Samples will be maintained frozen at approximately -20 °C for extended storage periods.

**Sample Processing** The following describes the preparation of samples for dicamba analysis by LC-MS/MS. A typical analytical set will include study samples, QCs and standards.

Step	Action										
1	Add a single PUF to each tube designated as a QC.										
2	Add 0.100 mL of the following solution to the designated sample type: <ul style="list-style-type: none"> <li>• ACN to test samples and controls</li> <li>• Working Calibration Solution to calibration standards</li> <li>• QC Fortification Solution to QC samples (e.g. LOQ QC, Mid QC, High QC and/or dilution QC)</li> </ul> <table border="1"> <thead> <tr> <th>QC Sample</th><th>Fortification Level (ng/PUF)</th></tr> </thead> <tbody> <tr> <td>LOQ QC</td><td>0.10</td></tr> <tr> <td>Mid QC</td><td>1.0</td></tr> <tr> <td>High QC</td><td>6.0</td></tr> <tr> <td>Dilution QC</td><td>60</td></tr> </tbody> </table>	QC Sample	Fortification Level (ng/PUF)	LOQ QC	0.10	Mid QC	1.0	High QC	6.0	Dilution QC	60
QC Sample	Fortification Level (ng/PUF)										
LOQ QC	0.10										
Mid QC	1.0										
High QC	6.0										
Dilution QC	60										
3	Add 0.100 mL of the IS Working Solution (0.03 µg/mL) to all tubes (including tubes designated as standards) using an automated liquid handler or other repeat pipetting device.										
4	Add two grinding balls to each tube.										
5	Add approximately 29.8 mL of MeOH to all tubes using a bottle-top dispenser or other repeat pipetting device.										
6	Place a cap on each tube. Ensure the cap is sealed well before proceeding.										
7	Shake samples on the Geno/Grinder® to extract analyte from the PUF (e.g., 1200 cycles per minute for 30 minutes). Examine the tubes for leaks. If leaks are detected, discard and re-prepare.										
8	Filter a portion (e.g., 11 mL) of extract using a 0.45µm PTFE syringe filter into a clean 15-mL polypropylene centrifuge tube sufficient to yield 10 mL of filtered extract.										
9	Aliquot 10 mL of filtered extract into clean 15-mL graduated polypropylene centrifuge tubes and evaporate to approximately 1 mL under a gentle stream of nitrogen using N-Evap with water bath set at ~45°C.										

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 10 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

10	Transfer concentrated extracts to clean 96-well long drip, polypropylene filter plate (0.45 µm) with a clean tapered well polypropylene plate (2 mL) positioned below the filter plate.  <b>Note:</b> Vortex and sonicate concentrated extracts prior to transfer to ensure full recovery.
11	Pass the samples through the filter plate using vacuum as needed.
12	Discard the filter plate. Evaporate the samples to dryness under nitrogen. <b>Note:</b> This step is performed at 50 °C. Typical setting: N <sub>2</sub> = 40 L/min.
13	Reconstitute samples with 0.100 mL of 25% methanol in water solution.
14	Cover the 96-well plate with a square well sealing cap mat. Ensure the cap mat is sealed well before proceeding.
15	Mix well using a multi-tube vortexer and analyze by LC-MS/MS within the storage time determined during method validation.

**Injection Volume Adjustment** High-level samples producing an analyte response greater than that of the highest standard of the calibration curve must be re-injected with a smaller injection volume (down to 1 µL) so the analyte response of the sample is within the analyte response range of the standards. This procedure maintains the response ratio. It is not necessary to enter a dilution factor in the calculations. **Note:** The response ratio may not be within the range of response ratios of the standards. A “dilution QC” must be included and pass acceptance criteria to accept small injection volume data. The re-injected sample may be analyzed in any chromatographic set.

If multiple injections of a study sample are performed, the first acceptable result will be reported.

### Instrumental Analysis

<b>Sample Analysis Guidance and Acceptance Criteria</b>	<p>An analytical set contains calibration standards, controls, QCs, and test samples. Each set must contain a minimum of duplicate control samples and triplicate QC fortifications at the LOQ and triplicate fortifications at least one higher QC level. The chromatographic batch must begin and end with a calibration standard (i.e., controls, QCs, and unknown samples are bracketed by calibration standards).</p> <ul style="list-style-type: none"><li>• Analyte calibration must be performed for each set using a calibration curve with a minimum of six concentration levels (excluding blanks) and at least 75% of the total number of calibration standards represented in the final curve. The final curve must have a correlation coefficient (<math>r \geq 0.995</math> (<math>r^2 \geq 0.99</math>)).</li><li>• Calibration standard concentrations used to determine results must be within <math>\pm 20\%</math> of their respective nominal concentrations (i.e., accuracy is 80-120%) when back-calculated against the calibration curve.</li><li>• Calibration points may be removed for a documented analytical reason (e.g. fails an outlier test) or a calculated inaccuracy outside <math>\pm 20\%</math>. If a calibration standard(s) is removed the reason must be documented in the raw data (e.g., inaccuracy <math>&gt; 20\%</math>).</li><li>• QC fortifications must have a mean accuracy within 70-120% of the nominal value and a precision of <math>\leq 20\%</math> RSD at each QC level.</li><li>• The average calculated concentration in QC controls compared to the nominal LOQ must have a ratio <math>\leq 30\%</math> to demonstrate acceptable selectivity. In cases where this response is exceeded, the presence of the target (i.e. inadvertent contamination) versus an unknown interference will be assessed using an appropriate confirmatory technique.</li><li>• The potential for carryover will be evaluated in each analytical or batch run by placing a double blank after the highest calibration standard. The response for analyte in the carryover sample must be <math>\leq 20\%</math> of the response at the LOQ level. In cases where this is exceeded the data will be evaluated by the analytical PI and the Study Director to determine the potential impact on the study and, if needed, corrective actions to ensure accurate measurements are attained.</li><li>• If low volume injection is needed, then capability of low volume injection will be demonstrated by including dilution QC samples in the study. The dilution QC samples must meet the same acceptance criteria for accuracy and precision for quality control samples as stated above.</li><li>• Acceptance of data that do not meet the above criteria must have a documented reason and approval by the Study Director.</li></ul>
---	---

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 12 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Instrument Setup**

Instrument operation is controlled by acquisition methods containing all autosampler, HPLC, switching valve, source interface and mass spectrometer parameters. Precursor and product ions for the analytes are shown below along with choices for possible use in confirmatory analyses. Alternate ions may be used for quantitation or confirmation if they provide better data (sensitivity and/or specificity). The use of a minimum of one quantitation transition and one confirmatory transition is required for each batch run. The transitions used for these purposes will be clearly designated. The following equipment and conditions are instrument/system dependent and may be modified to obtain optimal instrument performance and maximize sensitivity. Injection volume may be modified to extend the dynamic range of the method. Actual method parameters must be documented in the raw data.

**System Conditions for Analysis**

**LC-MS/MS System Conditions**

HPLC: Shimadzu ExionLC AD  
Mass spectrometer: AB Sciex API 6500+ with SelexION®+  
Ion source: Turbo-V  
Column: Phenomenex Kinetex Biphenyl Column, 50 mm × 3.0 mm, 2.6 µm  
Injection volume: 10 µL  
Autosampler temperature: 10 °C  
Column oven temperature: 40 °C  
Mobile Phase A: 0.05% formic acid (aq)  
Mobile Phase B: MeOH

**HPLC Gradient Conditions:**

Time (min)	%B	Total Flow (mL/min)	Divert
0	20	0.5	To waste
2.0	20	0.5	To MS
4.0	55	0.5	To MS
4.01	95	0.5	To MS
4.8	95	0.5	To waste
6.0	95	0.5	To waste
6.01	20	0.5	To waste
7.0	Controller Stop		

Run time: 7 min (MS data collection 5 min)

**Mass Spectrometer Conditions**

Mode: negative ion  
Scan type: MRM  
Resolution Q1: unit  
Resolution Q3: low  
Probe type: ESI

Duration : 5 min Curtain gas (CUR): 20 Collision gas (CAD): Medium Gas 1: 70 N <sub>2</sub> Gas 2: 60 N <sub>2</sub>	IonSpray voltage (IS): -2500 V Interface heater: on Temperature (TEM): 300 °C Scan time (ms): 250
--	--

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 13 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

Analyte:	Precursor Ion Q1 (amu)	Product Ion Q3 (amu)	DP (V)	EP (V)	CE (V)	CXP (V)
Dicamba	219	175	-10	-10	-9	-9
<sup>13</sup> C <sub>6</sub> )Dicamba (IS)	225	181	-10	-10	-9	-9
Confirmatory Ions						
Dicamba	221	177	-10	-10	-9	-9
<sup>13</sup> C <sub>6</sub> )Dicamba (IS)	227	183	-10	-10	-9	-9
SelexION+ Conditions						
Separation Voltage (SV): 2000 Compensation Voltage (COV): -11 DMS Offset (DMO): 50 DMS Temperature (DT): Low DMS Resolution Enhancement (DR): Open						
Modifier: 2-propanol Modifier Compensation (MDC): low Modifier Density (MDD): 0.79 Modifier MW (MDW): 60.10						

**Data Processing**

Process the data using the Analyst® quantitation wizard. The wizard is used to process the data for the MRM transition pairs established in the acquisition method. The method detects and integrates the analyte peaks based on retention time and MRM transition. Chromatograms may be smoothed prior to integration as long as the smoothing is consistent throughout the entire chromatographic set. Manual peak integration should be used when the automated procedure is not effective due to baseline noise. Dilution factors, if applicable, must be added during data processing if not input prior to the start of the instrument run.

**Calculations**

**Overview**

Analyte concentrations are calculated using the Analyst® software. The software calculates the standard curve and applies the dilution factor to account for dilution or concentration during processing. Standard curves are generated as the ratio of the analyte response (e.g., peak area) to the internal standard response, for each standard level, plotted against concentration (i.e., ratio of analyte concentration to internal standard concentration). A linear regression model is used for quantitation with or without weighting (e.g. linear 1/x weighted). All the samples from a study must be analyzed with the same type of calibration curve (i.e., plot axes and weighting) for a given analyte transition.

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 14 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps**

**Analyte Concentration**      Analyte concentrations are reported as ng/PUF. The Analyst® software automatically calculates the raw concentration of the injected sample relative to the standard curve (*calculated concentration*). This value is also automatically multiplied by any value entered in the *dilution factor* column.

**Assumptions:**

- 1) The nominal dilution of the sample during extraction (1:30) is incorporated into the calibration standard concentrations that are entered into the Analyst® software. The calibration standard concentrations are entered as equivalent concentrations ('Equivalent Conc. (ng/PUF)' in the Working Calibration Solution tables above). Calibration standard solutions are diluted equivalently to samples in the sample processing procedure of the method; therefore, these entered concentrations are 30 times their actual injected concentrations, so the dilution factor is eliminated.
- 2) Entry of a dilution factor is not required for samples using a reduced injection volume or extract concentration (the original analyte/IS response ratio is maintained).

**Documentation**

The analytical raw data packages will include (at a minimum): the sample processing worksheet (form(s)), instrumental sample queue/run record, calibration curves, MRM chromatograms, results tables, and instrument acquisition parameters.

**Example Chromatograms**

Example chromatograms for calibration standards, control samples and fortified samples are provided in Appendix B.

**Method Validation Results**

Method Validation Summary is provided in Appendix C.

Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 15 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

Superseded method(s): N/A

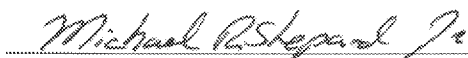
Author:

  
Leah Riter

Date July 11 2019

(Analytical Methods and Analysis Lead, Monsanto Company)

Management:

  
Michael R. Shepard

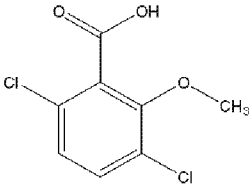
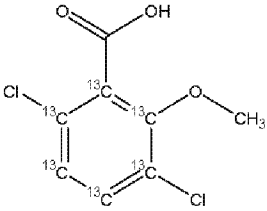
Date July 11 2019

(Structural Elucidation & Residue Analysis Lead, Monsanto Company)



## Appendices

### Appendix A: Compound Structures

	<p>Dicamba</p> <p>3,6-dichloro-2-methoxybenzoic acid</p> <p>CAS # 1918-00-9</p> <p><math>C_8H_6Cl_2O_3</math></p> <p>Average Molecular weight: 221.04</p>
	<p><math>(^{13}C_6)</math>Dicamba</p> <p>3,6-dichloro-2-methoxybenzoic-1,2,3,4,5,6-<math>^{13}C_6</math> acid</p> <p>CAS #1173023-06-7</p> <p><math>C_2^{13}C_6H_4Cl_2O_3</math></p> <p>Average Molecular weight: 227.04</p>

Monsanto Company Method

Effective Date: July 05, 2019

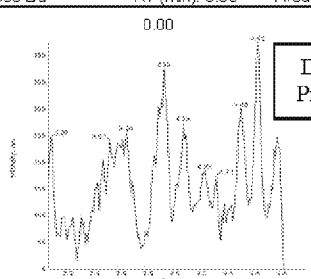
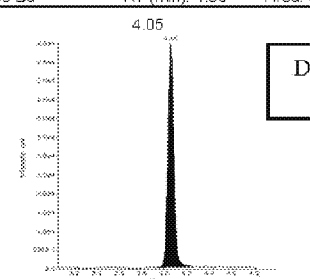
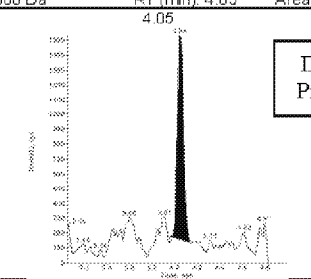
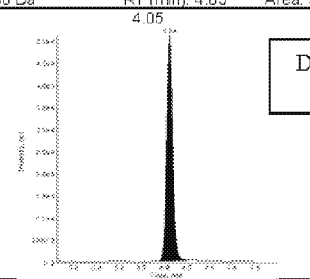
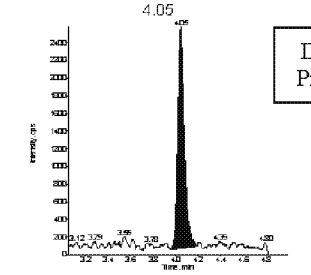
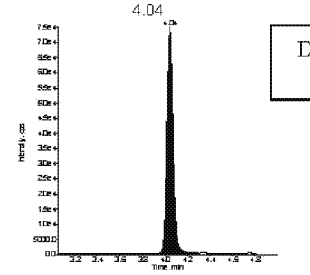
ME-2242-01

Page 17 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

Appendix B: Example Chromatograms

Dicamba primary quantitation ion transition in PUF

Sample ID	PUF Control	Sample Name	88605-MV01
Sample Type	Quality Control	Sample Comment	QC Control
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 219.000/175.000 Da RT (min): 0.00 Area: 0.00e+00		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.05 Area: 6.06e+04	
			
Sample ID	PUF Fort. + 0.100 ng/PUF	Sample Name	88605-MV03
Sample Type	Quality Control	Sample Comment	LOQ QC
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 219.000/175.000 Da RT (min): 4.05 Area: 1.41e+03		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.05 Area: 5.19e+04	
			
Sample ID	0.100 ng/PUF	Sample Name	88605-ST047
Sample Type	Standard	Sample Comment	0.00100 µg/mL
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 219.000/175.000 Da RT (min): 4.05 Area: 2.54e+03		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.04 Area: 7.55e+04	
			

.Dicamba Primary 1: untreated control (left); IS (right)

Dicamba Primary 2: fortified control 0.10 ng/PUF; IS (right)

Dicamba Primary 3: calibration standard 0.10 ng/PUF; IS (right)

Monsanto Company Method

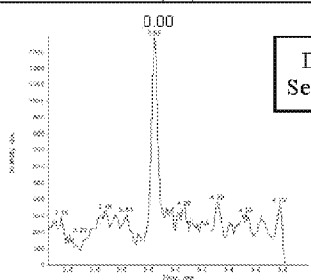
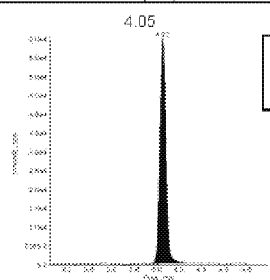
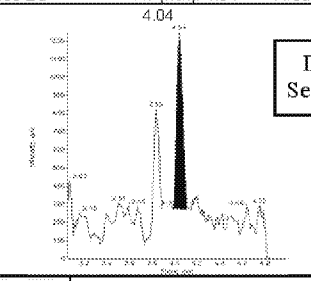
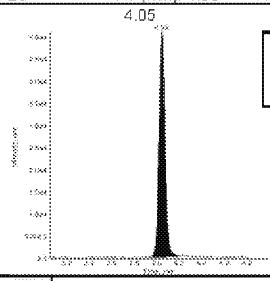
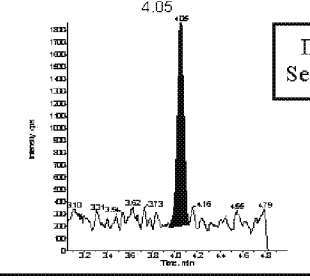
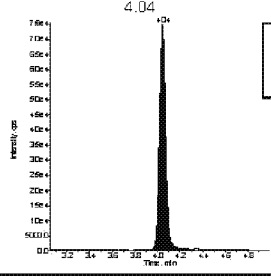
Effective Date: July 05, 2019

ME-2242-01

Page 18 of 21

LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps

Dicamba secondary quantitation ion transition in PUF

Sample ID	PUF Control	Sample Name	88605-MV01
Sample Type	Quality Control	Sample Comment	QC Control
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 221.000/177.000 Da RT (min): 0.00 Area: 0.00e+00		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.05 Area: 6.06e+04	
 <p>Dicamba Secondary 1</p>		 <p>Dicamba IS 1</p>	
Sample ID	PUF Fort. + 0.100 ng/PUF	Sample Name	88605-MV03
Sample Type	Quality Control	Sample Comment	LOQ QC
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 221.000/177.000 Da RT (min): 4.04 Area: 9.80e+02		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.05 Area: 5.19e+04	
 <p>Dicamba Secondary 2</p>		 <p>Dicamba IS 2</p>	
Sample ID	0.100 ng/PUF	Sample Name	88605-ST047
Sample Type	Standard	Sample Comment	0.00100 µg/mL
Results File Name	C:\5064_88605_04292019A.rdb		
Analyte: Dicamba 221.000/177.000 Da RT (min): 4.05 Area: 1.66e+03		Internal Standard: Dicamba_IS 225.000/181.000 Da RT (min): 4.04 Area: 7.55e+04	
 <p>Dicamba Secondary 3</p>		 <p>Dicamba IS 3</p>	

.Dicamba Secondary 1: untreated control (left); IS (right)  
Dicamba Secondary 2: fortified control 0.10 ng/PUF; IS (right)  
Dicamba Secondary 3: calibration standard 0.10 ng/PUF; IS (right)

### **Appendix C: Validation Summary**

Report:	Development and Validation for the Analytical Method ME 2242: LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps, Authority registration No.: N/A
Guideline(s):	The method complies with the standard acceptance criteria of EPA Guideline OPPTS 860.1340 – Residue Analytical Method.
Deviations:	Yes
GLP:	Yes

### **Principle of the Method**

Dicamba was extracted from polyurethane foam (PUF) air sampling traps using methanol containing stable-labelled internal standard. The sample tubes were capped and agitated on a high-speed shaker for extraction. A 10-mL aliquot of the supernatant was filtered, evaporated to approximately 10% of its original volume, then re-filtered and evaporated to dryness. The samples were reconstituted in 0.10 mL of 25% methanol in water, yielding a 100-fold concentration factor.

Dicamba was quantitated using LC-MS/MS with electrospray ionization in negative ion mode with SelexION®+ Differential Mobility Separation. The working range of the method was from 0.0300 to 7.50 ng/PUF (0.100 to 25.0 ng/mL injected on column), with an LOQ of 0.10 ng/PUF. Extension of the upper range of the method using lower injection volumes was demonstrated during method validation. Method performance and recovery data of dicamba from polyurethane foam (PUF) air sampling traps are presented in this summary.

### **Results and Discussions**

#### **Recovery Findings**

The recovery results for dicamba are presented in the tables below. Results obtained are within guideline EPA Guideline OPPTS 860.1340 – Residue Analytical Method (Mean recovery 70-120%; RSD ≤ 20%).

#### **Conclusion**

The data obtained during this method validation for the determination of dicamba in PUF air sampling traps met the acceptability criteria established in the protocol and demonstrated satisfactory results in terms of accuracy, precision, selectivity, linearity, and specificity based on the quantitation and confirmation transitions monitored for PUF samples fortified at the proposed LOQ (0.10 ng/PUF) and at 10, 60, and 600 times the proposed LOQ (1.0, 6.0, and 60 ng/PUF, respectively). Additionally, results obtained from reinjecting the final extracts indicate that residues of dicamba are stable in PUFs when stored refrigerated (2 to 8 °C) for up to seven days.

## Monsanto Company Method

Effective Date: July 05, 2019

ME-2242-01

Page 20 of 21

**LC-MS/MS Method for Quantitation of Dicamba in Polyurethane Foam (PUF) Air Sampling Traps****Table 1: Recovery Results from Method Validation of Dicamba in PUF Air Sampling Traps Using the Analytical Method ME-2242-01**

Matrix	Fortification Level [ng/PUF]	Range of Recoveries [%-%]	Mean [%]	Std. Dev. [%]	Rel. Std. Dev. [%]	No. of Analyses
Dicamba, MRM $m/z$ 219→175 (quantitation)						
PUF	0.100	88-103	99	5.2	5.3	7
	1.00	88-97	92	3.8	4.2	6
	6.00	88-95	93	2.6	2.8	6
	60.0	97-105	100	3.4	3.4	6
	<b>Overall</b>	<b>88-105</b>	<b>96</b>	<b>5.2</b>	<b>5.4</b>	<b>25</b>
Dicamba, MRM $m/z$ 221→177 (confirmation)						
PUF	0.100	92-118	105	10.0	9.5	7
	1.00	94-99	96	1.9	2.0	6
	6.00	90-97	94	2.4	2.5	6
	60.0	96-106	101	3.7	3.7	6
	<b>Overall</b>	<b>90-118</b>	<b>99</b>	<b>7.0</b>	<b>7.1</b>	<b>25</b>

**Table 2: Characteristics for the Analytical Method Used for Validation of Dicamba Residues in PUF Air Sampling Traps**

	Dicamba
Specificity and selectivity	Analysis of control specimens of PUF with HPLC-MS/MS using at least two mass transitions yielded no mean residues of dicamba above 30% of the LOQ indicating that no significant interferences were present. At least two mass transitions were monitored: $m/z$ 219 → 175 (quantitation) $m/z$ 221 → 177 (confirmation)
Calibration (type, number of data points)	The linearity of the detector response was demonstrated by single determination of calibration standards at 10 concentration levels ranging from 0.0300 to 7.50 ng/PUF of dicamba. Calibration solutions also contained the isotopically enriched internal standard $^{13}\text{C}$ dicamba. The concentration ranges covered at least 30% of the LOQ to at least 20% above the 10 x LOQ equivalent standard concentration level detected in a sample. The coefficient of determination ( $r^2$ ) was $\geq 0.99$ for all analytical determinations.
Assessment of matrix effects is presented	It has been demonstrated in this validation that the internal standard compensates for the matrix effects based on acceptable recovery of fortification samples using non matrix (solvent) calibration curve standards.
Stability of sample extracts	Dicamba was tested to be stable in final extracts for at least 7 days when stored refrigerated (2 to 8 °C).
Limit of quantitation (LOQ)	LOQ is 0.10 ng/PUF
Limit of detection (LOD)	LOD is 0.030 ng/PUF

Figure 1: Typical Standard Calibration Plot for Dicamba Primary Quantitation Ion Transition

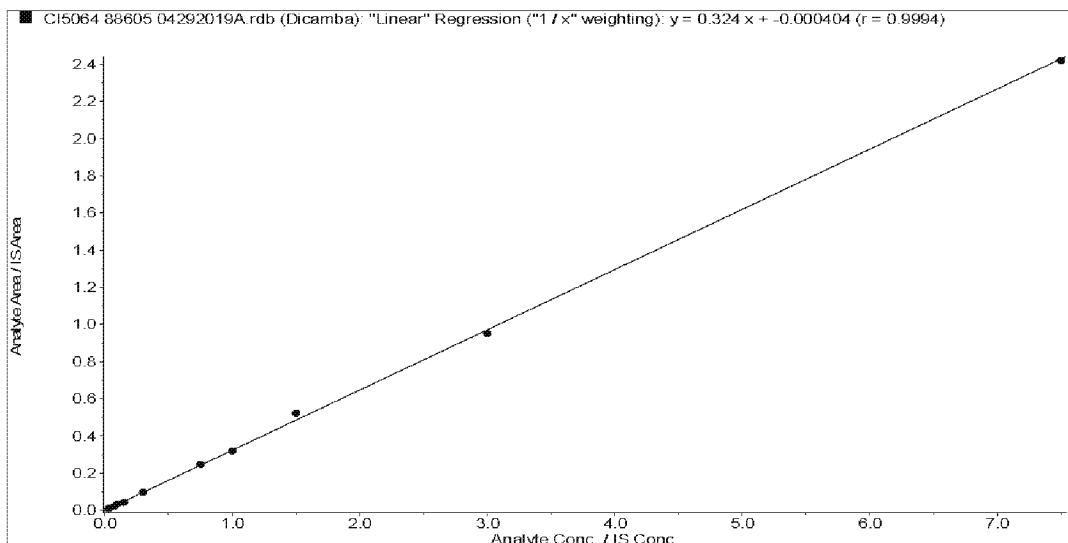
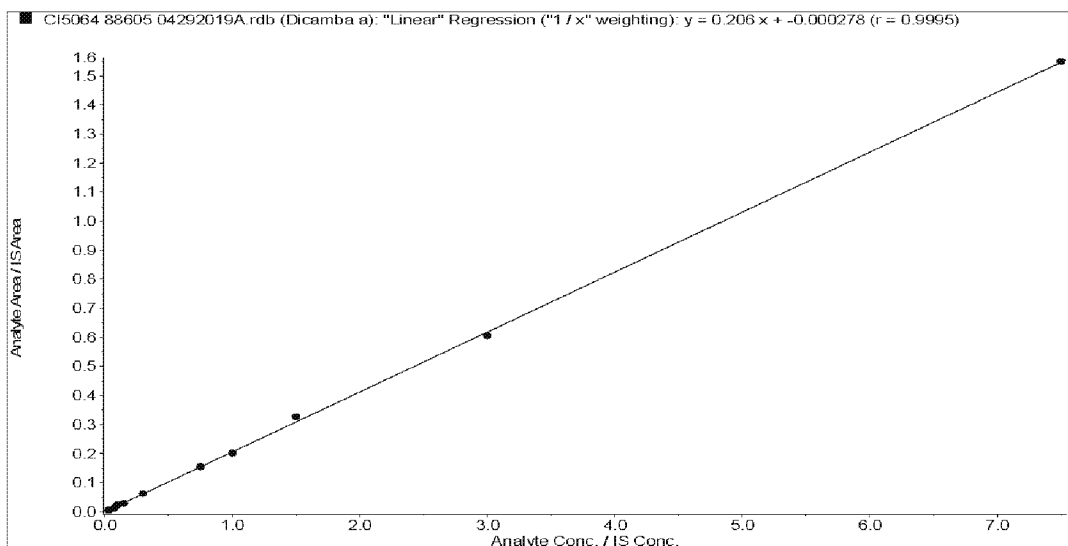


Figure 2: Typical Standard Calibration Plot for Dicamba Secondary Quantitation Ion Transition



## **Appendix E. Raw Data, Recoveries, and Statistics for Application Monitoring Filter Paper Analysis**

This appendix contains the following tables which present data relevant to this report:

- E.1 Recovery Data Table and Statistics for Application Monitoring Filter Paper  
Samples Fortified with Dicamba
- E.2 Raw Data Table for Application Monitoring Filter Paper Sample Analysis

### **E.1 Recovery Data Table and Statistics for Application Monitoring Filter Paper Samples Fortified with Dicamba**

The *Recovery Data Table for Application Monitoring Filter Paper Samples Fortified with Dicamba* contains the data on the recovery of fortified samples analyzed in the same analytical sets as treated samples. These data were used to generate result summary [Table 1](#). The description of each column is presented below. Statistical data follow the recovery table.

#### **A. EEAL ID**

The analytical ID used to track the sample through analysis.

#### **B. Set No.**

Analytical set number

#### **C. Extraction Date**

The date the samples were extracted in the analytical laboratory.

#### **D. Analysis Date**

The date the analytical results were acquired from the instrument.

#### **E. Peak Area**

Area of the analyte as determined by Empower.

#### **F. Curve Constants (m and b)**

Calculations for instrumental analysis were conducted using a validated software application (e.g., Empower 3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve ( $1/x^2$ ) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$

where:

y = peak area of the analyte

m = slope of curve

x = detector response in  $\mu\text{g/g}$

b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x (\mu\text{g/g}) = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery



data tables. The amounts of dicamba found presented here were calculated based on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

**G. Dose Sol. Conc. (mg/g)**

The amount of dicamba in mg/g in the fortification solution.

**H. Wt. of Dose Sol. (g)**

Weight of the fortification (dosing) solution in grams.

**I. Wt. of Water (g)**

Weight of water in grams.

**J. Theor. Conc. (mg)**

The theoretical amount of analyte used to spike the filter paper sample. The nominal concentration in mg was calculated according to the following equation:

$$\text{Theor. Conc. (mg)} = \text{Dose Sol. Conc. (mg/g)} \times \text{Wt. of Dose Sol. (g)}$$

**K. Meas. Conc. (mg)**

The amount of analyte in mg of analyte recovered from the fortified sample. The amount of analyte determined as mg per filter paper was calculated according to the following equation:

$$\text{Meas. Conc. (mg)} = \frac{\mu\text{g/g Found} \times \text{Wt. of Water (g)}}{1000}$$

**L. Fort. Level**

The fortification level of each sample expressed as either “Low” or “High”.

**M. Net % Rec.**

The percent of the analyte recovered from the fortified sample calculated according to the following equation.

$$\text{Net \% Rec.} = \frac{\text{Meas. Conc. (mg)}}{\text{Theor. Conc. (mg)}} \times 100$$

## N. Example Calculation – Fortified Sample

From the Raw Data Table:

EEAL/Sample ID: FP-QC-L-1-F

Sample Description: Filter paper, low-level fortification

Analytical Set: Filter Paper 18Jul19

where:

Dose Sol. Conc. = 19.7 mg/g

Wt. of Dose Sol. = 0.02738 g

peak area (y) = 64498

y-intercept (b) = 356

slope (m) = 3470

Wt. of Water = 29.91937 g

1000 = conversion from µg to mg

$$\text{Theor. Conc. (mg)} = 19.7 \text{ mg/g} \times 0.02738 \text{ g} = 0.539 \text{ mg}$$

$$\text{Meas. Con. (mg)} = \frac{\frac{(64498 - 356)}{3470} \times 29.91937 \text{ g}}{1000} = 0.553 \text{ mg}$$

$$\text{Net \% Rec very} = \frac{0.553 \text{ mg}}{0.539 \text{ mg}} \times 100 = 103\%$$

## Statistics

The statistical methods used in this study were limited to calculations of the mean, range, standard deviation, and relative standard deviation. Statistics were calculated using rounded percent recovery values. Microsoft Excel® 2016 was employed to develop all statistical data.

### Recovery Data Table for Application Monitoring Filter Paper Samples Fortified with Dicamba

EEAL ID	Set No.	Extraction Date	Analysis Date	Peak Area	Curve Constants		Dose Sol. Conc. (mg/g)	Wt. of Dose Sol. (g)	Wt. of Water (g)	Theor. Conc. (mg)	Meas. Conc. (mg)	Fort. Level	Net % Rec.
					m	b							
FP-QC-L-1-F	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	64498	3470	356	19.7	0.02738	29.91937	0.539	0.553	Low	103
FP-QC-L-2-F	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	65469	3470	356	19.7	0.02735	29.86184	0.539	0.560	Low	104
FP-QC-L-3-F	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	65582	3470	356	19.7	0.02745	29.84326	0.541	0.561	Low	104
FP-QC-H-1-F	Filter Paper 18Jul19	18-Jul-19	19-Jul-19	108970	3470	356	19.7	0.04595	29.84440	0.905	0.934	High	103
FP-QC-H-2-F	Filter Paper 18Jul19	18-Jul-19	19-Jul-19	108441	3470	356	19.7	0.04579	29.91318	0.902	0.932	High	103
FP-QC-H-3-F	Filter Paper 18Jul19	18-Jul-19	19-Jul-19	108617	3470	356	19.7	0.04587	29.84562	0.904	0.931	High	103

### Recovery Statistics for Application Monitoring Filter Paper Samples Fortified with Dicamba

	Low	High
Number of Fortifications	3	3
Minimum Recovery (%)	103	103
Maximum Recovery (%)	104	103
Mean (%)	104	103
Standard Deviation (%)	0.6	0.0
Relative Standard Deviation (%)	0.6	0.0

## **E.2 Raw Data Table for Application Monitoring Filter Paper Sample Analysis**

The *Raw Data Table for Application Monitoring Filter Paper Sample Analysis* contains data from which result summary Table 2 was generated. The description of each column is presented below.

### **A. EEAL ID**

The analytical ID used to track the sample through analysis.

### **B. Sample ID**

The field ID which identifies study, sample type, location, timing, and replicate for each sample analyzed.

### **C. Set No.**

Analytical set number

### **D. Extraction Date**

The date the samples were extracted in the analytical laboratory.

### **E. Analysis Date**

The date the analytical results were acquired from the instrument.

### **F. Peak Area**

Area of the analyte as determined by Empower.

### **G. Curve Constants (m and b)**

Calculations for instrumental analysis were conducted using a validated software application (e.g., Empower 3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve ( $1/x^2$ ) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$

where:

y = peak area of the analyte

m = slope of curve

x = detector response in  $\mu\text{g/g}$

b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x (\mu\text{g/g}) = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery data tables. The amounts of dicamba found presented here were calculated based on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

#### H. Wt. of Water (g)

Weight of water in grams.

#### I. Dicamba (mg/filter)

The amount of analyte found in mg of analyte per filter paper sample collector as calculated according to the following equation:

$$\text{Dicamba (mg/filter)} = \frac{\mu\text{g/g Found} \times \text{Wt. of Water (g)}}{1000}$$

#### J. Example Calculation – Treated Sample

From the Raw Data Table:

EEAL ID: 496194-F

Sample ID: REG-2019-0035 . FPS . A . APP . 2 . 004

Analytical Set: Filter Paper 18Jul19

where:

peak area (y) = 85175

y-intercept (b) = 356

slope (m) = 3470

wt. of water = 29.84364 g

1000 = conversion from  $\mu\text{g}$  to mg

$$\text{Dicamba (mg/filter)} = \frac{(85175 - 356)}{3470} \times 29.84364 \text{ g} = 0.729 \text{ mg/filter}$$

**Raw Data Table for Application Monitoring Filter Paper Sample Analysis**

EEAL ID	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	Curve Constants		Wt. of Water (g)	Dicamba (mg/filter)
						m	b		
496193-F	REG-2019-0035 . FPS . A . APP . 1 . 003	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	78972	3470	356	29.87658	0.677
496194-F	REG-2019-0035 . FPS . A . APP . 2 . 004	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	85175	3470	356	29.84364	0.729
496195-F	REG-2019-0035 . FPS . A . APP . 3 . 005	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	85412	3470	356	29.84571	0.732
496196-F	REG-2019-0035 . FPS . A . APP . 4 . 006	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	75887	3470	356	29.86542	0.650
496197-F	REG-2019-0035 . FPS . B . APP . 1 . 007	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	77117	3470	356	29.83536	0.660
496198-F	REG-2019-0035 . FPS . B . APP . 2 . 008	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	67714	3470	356	29.84716	0.579
496199-F	REG-2019-0035 . FPS . B . APP . 3 . 009	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	78871	3470	356	29.87939	0.676
496200-F	REG-2019-0035 . FPS . B . APP . 4 . 010	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	75218	3470	356	29.86052	0.644
496201-F	REG-2019-0035 . FPS . C . APP . 1 . 011	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	72233	3470	356	29.88305	0.619
496202-F	REG-2019-0035 . FPS . C . APP . 2 . 012	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	75468	3470	356	29.87371	0.647
496203-F	REG-2019-0035 . FPS . C . APP . 3 . 013	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	59429	3470	356	29.84179	0.508
496204-F	REG-2019-0035 . FPS . C . APP . 4 . 014	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	64730	3470	356	29.87304	0.554
496205-F	REG-2019-0035 . FPS . D . APP . 1 . 015	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	70598	3470	356	29.78569	0.603
496206-F	REG-2019-0035 . FPS . D . APP . 2 . 016	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	72540	3470	356	29.86072	0.621
496207-F	REG-2019-0035 . FPS . D . APP . 3 . 017	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	73045	3470	356	29.83569	0.625
496208-F	REG-2019-0035 . FPS . D . APP . 4 . 018	Filter Paper 18Jul19	18-Jul-19	18-Jul-19	66679	3470	356	29.87896	0.571

## **Appendix F. Raw Data, Recoveries, and Statistics for Tank Mix Sample Analysis**

This appendix contains the following tables which present data relevant to this report:

- F.1 Recovery Data and Statistics for Tank Mix Samples Fortified with Dicamba
- F.2 Raw Data Table for Tank Mix Sample Analysis

### F.1 Recovery Data and Statistics for Tank Mix Samples Fortified with Dicamba

The *Recovery Data Table for Tank Mix Samples Fortified with Dicamba* contains the data on the recovery of fortified samples analyzed in the same analytical sets as treated samples. These data were used to generate result summary Table 3. The description of each column is presented below. Statistical data follow the recovery table.

#### A. EEAL ID

The analytical ID used to track the sample through analysis.

#### B. Set No.

Analytical set number

#### C. Extraction Date

The date the samples were extracted in the analytical laboratory.

#### D. Analysis Date

The date the analytical results were acquired from the instrument.

#### E. Peak Area

Area of the analyte as determined by Empower.

#### F. Curve Constants (m and b)

Calculations for instrumental analysis were conducted using a validated software application (e.g., Empower 3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve ( $1/x^2$ ) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$

where:

y = peak area of the analyte

m = slope of curve

x = detector response in  $\mu\text{g/g}$

b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x (\mu\text{g/g}) = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery data tables. The amounts of dicamba found presented here were calculated based



on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

**G. Conc. from Empower (µg/g)**

Dicamba result in µg/g obtained from Empower.

**H. Purity of Form. (%)**

The amount of dicamba in the formulation used to fortify quality control samples.

**I. Wt. of Form. (g)**

Weight of the fortification formulation in grams.

**J. Wt. of Water (g)**

Weight of water in grams.

**K. Wt. of Aliq. (g)**

Weight of the dilution aliquot.

**L. Wt. of Diluent (g)**

Weight of the dilution diluent.

**M. Theor. Conc. (g/g)**

The theoretical amount of analyte used to spike the tank mix sample. The nominal concentration in g/g was calculated according to the following equation:

$$\text{Theor. Conc. (g/g)} = \frac{\text{Purity of Form. \%} \times \text{Wt. of Form. (g)}}{100 \times (\text{Wt. of Form. (g)} + \text{Wt. of Water (g)})}$$

**N. Final Conc. (g/g)**

The amount of analyte determined as g/g was calculated according to the following equation:

$$\text{Final Conc. (g/g)} =$$

$$\frac{\text{Con. from Empower (µg/g)} \times (\text{Wt. of Aliq. (g)} + \text{Wt. of Diluent (g)})}{\text{Wt. of Aliq. (g)} \times 1000000}$$

**O. Fort. Level**

The fortification level of each sample expressed as either “Low” or “High”.

**P. Net % Rec.**

The percent of the analyte recovered from the fortified sample calculated according to the following equation.

$$\text{Net \% Rec.} = \frac{\text{Final Conc. (g/g)}}{\text{Theor. Conc. (g/g)}} \times 100$$

## Q. Example Calculation – Fortified Tank Mix Sample

From the Raw Data Table:

EEAL/Sample ID: 89311-QC-L-1-D  
Sample Description: Tank mix, low-level fortification  
Analytical Set: Tank Mix 19Jul19

where:

peak area (y) = 258942  
y-intercept (b) = 347  
slope (m) = 3470  
Purity of Form. = 9.84% dicamba  
Wt. of Form. = 0.50408 g  
Wt. of Water = 16.00291 g  
Wt. of Aliq. = 0.50069 g  
Wt. of Diluent = 19.50542 g  
1000000 = conversion from µg to g

$$\text{Conc. from Empower } (\mu\text{g dicamba/g}) = \frac{(258942 - 347)}{3470} = 74.5 \mu\text{g dicamba/g}$$

$$\text{Theor. Con. } (g/g) = \frac{9.84 \% \times 0.50408 \text{ g}}{100 \times (0.50408 \text{ g} + 16.00291 \text{ g})} = 0.00300 \text{ g/g}$$

$$\text{Eln Con. } (g/g) = \frac{74.5 \mu\text{g/g} \times (0.50069 \text{ g} + 19.50542 \text{ g})}{0.50069 \text{ g} \times 1000000} = 0.00298 \text{ g/g}$$

$$\text{Net \% Rec very} = \frac{0.00298}{0.00300} \times 100 = 99\%$$

## Statistics

The statistical methods used in this study were limited to calculations of the mean, range, standard deviation, and relative standard deviation. Statistics were calculated using rounded percent recovery values. Microsoft Excel® 2016 was employed to develop all statistical data.

### Recovery Data Table for Tank Mix Samples Fortified with Dicamba

EEAL ID (89311-)	Set No.	Extraction Date	Analysis Date	Peak Area	Curve Constants		Conc. from Empower (µg/g)	Purity of Form. (%)	Wt. of Form. (g)	Wt. of Water (g)	Wt. of Aliq. (g)	Wt. of Diluent (g)	Theor. Conc. (g/g)	Final Conc. (g/g)	Fort. Level	Net % Rec.
					m	b										
QC-L-1-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	258942	3470	347	74.502812	9.84	0.50408	16.00291	0.50069	19.50542	0.00300	0.00298	Low	99
QC-L-2-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	259761	3470	347	74.738642	9.84	0.50162	16.00450	0.50510	19.50436	0.00299	0.00296	Low	99
QC-L-3-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	257472	3470	347	74.079129	9.84	0.50132	16.02882	0.50035	19.51523	0.00298	0.00296	Low	99
QC-H-1-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	431308	3470	347	124.162435	9.84	0.50038	9.31498	0.50012	19.50374	0.00502	0.00497	High	99
QC-H-2-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	437968	3470	347	126.081180	9.84	0.50173	9.30454	0.50429	19.51081	0.00503	0.00500	High	99
QC-H-3-D	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	439375	3470	347	126.486712	9.84	0.50873	9.31743	0.50102	19.50813	0.00509	0.00505	High	99

### Recovery Statistics for Tank Mix Samples Fortified with Dicamba

	Low	High
Number of Fortifications	3	3
Minimum Recovery (%)	99	99
Maximum Recovery (%)	99	99
Mean (%)	99	99
Standard Deviation (%)	0.0	0.0
Relative Standard Deviation (%)	0.0	0.0

## F.2 Raw Data Table for Tank Mix Sample Analysis

The *Raw Data Table for Tank Mix Sample Analysis* contains data from which the result summary Table 4 was generated. The description of each column is presented below.

### A. EEAL ID

The analytical ID used to track the sample through analysis.

### B. Sample ID

The field ID which identifies study, sample type, timing, and replicate for each sample analyzed.

### C. Set No.

Analytical set number

### D. Extraction Date

The date the samples were extracted in the analytical laboratory.

### E. Analysis Date

The date the analytical results were acquired from the instrument.

### F. Peak Area

Area of the analyte as determined by Empower.

### G. Curve Constants (m and b)

Calculations for instrumental analysis were conducted using a validated software application (e.g., Empower 3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve ( $1/x^2$ ) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$

where:

y = peak area of the analyte

m = slope of curve

x = detector response in  $\mu\text{g/g}$

b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x (\mu\text{g/g}) = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery

data tables. The amounts of dicamba found presented here were calculated based on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

**H. Conc. from Empower (µg/g)**

Dicamba result in µg/g obtained from Empower.

**I. Wt. of Sample (g)**

Weight of sample in grams.

**J. Wt. of Water (g)**

Weight of water in grams.

**K. Dicamba (g/g)**

The amount of analyte found in g/g determined by the following equation:

$$\text{Dicamba (g/g)} = \frac{\text{Conc. from Empower (}\mu\text{g/g)} \times \frac{\text{Wt. of Sample (g)} + \text{Wt. of Water (g)}}{\text{Wt. of Sample (g)}}}{1000000}$$

**L. Dicamba (wt %)**

The amount of analyte found expressed as percentage.

**M. Example Calculation – Treated Sample**

From the Raw Data Table:

EEAL ID: 496209

Sample ID: REG-2019-0035 . TNK . A . PRE-APP . 1 . 019

Analytical Set: Tank Mix 19Jul19

where:

peak area (y) = 331664

y-intercept (b) = 347

slope (m) = 3470

wt. of sample = 0.50206 g

wt. of water = 19.50668 g

1000000 = conversion from µg to g

$$\text{Conc. from Empower (}\mu\text{g dicamba/g)} = \frac{(331664 - 347)}{3470} = 95.5 \mu\text{g dicamba/g}$$

$$\text{Dicamba (g/g)} = \frac{95.5 \mu\text{g/g} \times \left( \frac{0.50206 \text{ g} + 19.50668 \text{ g}}{0.50206 \text{ g}} \right)}{1000000} = 0.00380 \text{ g/g}$$

$$\text{Dicamba (wt\%)} = 0.380 \text{ wt\%}$$

### Raw Data Table for Tank Mix Sample Analysis

EEAL ID	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	Curve Constants		Conc. from Empower (µg/g)	Wt. of Sample (g)	Wt. of Water (g)	Dicamba (g/g)	Dicamba (wt%)
						m	b					
496209	REG-2019-0035 . TNK . A . PRE-APP . 1 . 019	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	331664	3470	347	95.454532	0.50206	19.50668	0.00380	0.380
496210	REG-2019-0035 . TNK . A . PRE-APP . 2 . 020	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	330632	3470	347	95.157170	0.50029	19.50710	0.00381	0.381
496211	REG-2019-0035 . TNK . A . PRE-APP . 3 . 021	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	333391	3470	347	95.951836	0.50298	19.51112	0.00382	0.382
496212	REG-2019-0035 . TNK . A . POST-APP . 1 . 022	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	329894	3470	347	94.944551	0.50033	19.50495	0.00380	0.380
496213	REG-2019-0035 . TNK . A . POST-APP . 2 . 023	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	335583	3470	347	96.583358	0.50852	19.51290	0.00380	0.380
496214	REG-2019-0035 . TNK . A . POST-APP . 3 . 024	Tank Mix 19Jul19	19-Jul-19	19-Jul-19	331760	3470	347	95.482079	0.50259	19.51389	0.00380	0.380

## **Appendix G. Raw Data, Recoveries, and Statistics for PUF Sample Analysis**

This appendix contains the following tables which present data relevant to this report:

- G.1 Recovery Data Table and Statistics for PUF Samples Fortified with Dicamba
- G.2 Raw Data Table for PUF Sample Analysis

### **G.1 Recovery Data Table and Statistics for PUF Samples Fortified with Dicamba**

The *Recovery Data Table for PUF Samples Fortified with Dicamba* contains the data on the recovery of fortified samples analyzed in the same analytical sets as treated samples. These data were used to generate recovery summary [Table 5](#). The description of each column is presented below. Statistical data follow the recovery table.

#### **A. EEAL ID**

The analytical ID used to track the sample through analysis.

#### **B. Sample Description**

Description of sample matrix and fortification level.

#### **C. Set No.**

Analytical set number

#### **D. Extraction Date**

The date the samples were extracted in the analytical laboratory.

#### **E. Analysis Date**

The date the analytical results were acquired from the instrument.

#### **F. Peak Area**

Peak area of the analyte

#### **G. IS Peak Area**

Peak area of the internal standard

#### **H. Area Ratio**

The ratio of the area of the analyte to the area of the internal standard determined according to the equation:

$$Area\ Ratio = \frac{Peak\ Area}{IS\ Peak\ Area}$$

#### **I. Curve Constants (m and b)**

Calculations for instrumental analysis were conducted using a validated software application (e.g., Analyst<sup>®</sup> 1.6.3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve (1/x) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$



where:

y = area ratio of the analyte  
m = slope of curve  
x = detector response in ng/PUF  
b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x \text{ (ng/PUF)} = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery data tables. The amounts of dicamba found presented here were calculated based on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

**J. Dicamba (ng/PUF)**

Dicamba residue found in ng of analyte per PUF sample collector obtained from Analyst<sup>®</sup>. Results corrected for the average control result are marked with an \*.

**K. Fort. Level (ng/PUF)**

The amount of analyte in ng added to untreated control PUF collectors.

**L. % Rec.**

The percent of the analyte recovered from the fortified sample. Net procedural recoveries from fortified samples were calculated according to the following equation:

$$\text{Net \% Rec} = \frac{\text{ng/PUF found} - \text{average ng/PUF found in control}}{\text{ng/PUF added}} \times 100$$

When no dicamba was detected in the procedural control samples, recoveries from fortified samples were calculated as follows:

$$\% \text{ Recovery} = \frac{\text{ng/PUF found}}{\text{ng/PUF added}} \times 100$$

### M. Example Calculation – Fortified Control PUF Sample

From the Raw Data Table:

EEAL ID: 89311-008

Sample Description: PUF Fort. + 6.00 ng/PUF

Analytical Set: 001

where:

peak area = 310947

IS peak area = 170398

area ratio (y) = 1.82483

y-intercept (b) = 0.00138691

slope (m) = 0.2965074

ng/PUF found in control = 0.01993, 0.00000

$$\text{Area Ratio} = \frac{310947}{170398} = 1.82483$$

$$\text{Dicamba (ng/PUF)} = \frac{(1.82483 - 0.00138691)}{0.2965074} = 6.15 \text{ ng/PUF}$$

$$\% \text{ Rec very} = \frac{6.15 \text{ ng/PUF} - \frac{(0.01993 + 0.00000)}{2}}{6.00 \text{ ng/PUF}} \times 100 = 102\%$$

### Statistics

The statistical methods used in this study were limited to calculations of the mean, range, standard deviation, and relative standard deviation. Statistics were calculated using rounded percent recovery values. Microsoft Excel® 2016 was employed to develop all statistical data.

**Recovery Data Table for PUF Samples Fortified with Dicamba**

EEAL ID (89311-)	Sample Description	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)	Fort. Level (ng/PUF)	% Rec.
								m	b			
001	PUF Control	001	15-Jul-19	16-Jul-19	0	182769	0.00000	0.2965074	0.00138691	0.00000	NA	NA
002	PUF Control	001	15-Jul-19	16-Jul-19	1279	175310	0.00730	0.2965074	0.00138691	0.01993	NA	NA
003	PUF Fort. + 0.100 ng/PUF	001	15-Jul-19	16-Jul-19	5309	173534	0.03060	0.2965074	0.00138691	0.08855*	0.100	89
004	PUF Fort. + 0.100 ng/PUF	001	15-Jul-19	16-Jul-19	6453	166084	0.03885	0.2965074	0.00138691	0.11640*	0.100	116
005	PUF Fort. + 0.100 ng/PUF	001	15-Jul-19	16-Jul-19	4667	169879	0.02747	0.2965074	0.00138691	0.07801*	0.100	78
006	PUF Fort. + 6.00 ng/PUF	001	15-Jul-19	16-Jul-19	315501	181712	1.73627	0.2965074	0.00138691	5.84111*	6.00	97
007	PUF Fort. + 6.00 ng/PUF	001	15-Jul-19	16-Jul-19	310791	185149	1.67860	0.2965074	0.00138691	5.64660*	6.00	94
008	PUF Fort. + 6.00 ng/PUF	001	15-Jul-19	16-Jul-19	310947	170398	1.82483	0.2965074	0.00138691	6.13977*	6.00	102
057	PUF Control	002	16-Jul-19	17-Jul-19	0	144317	0.00000	0.2935184	0.0008979839	0.00000	NA	NA
058	PUF Control	002	16-Jul-19	17-Jul-19	0	147259	0.00000	0.2935184	0.0008979839	0.00000	NA	NA
059	PUF Fort. + 0.100 ng/PUF	002	16-Jul-19	17-Jul-19	4242	139513	0.03041	0.2935184	0.0008979839	0.10053	0.100	101
060	PUF Fort. + 0.100 ng/PUF	002	16-Jul-19	17-Jul-19	4404	153339	0.02872	0.2935184	0.0008979839	0.09480	0.100	95
061	PUF Fort. + 0.100 ng/PUF	002	16-Jul-19	17-Jul-19	4227	134089	0.03153	0.2935184	0.0008979839	0.10435	0.100	104
062	PUF Fort. + 6.00 ng/PUF	002	16-Jul-19	17-Jul-19	255843	139018	1.84037	0.2935184	0.0008979839	6.26696	6.00	104
063	PUF Fort. + 6.00 ng/PUF	002	16-Jul-19	17-Jul-19	250024	135611	1.84369	0.2935184	0.0008979839	6.27828	6.00	105
064	PUF Fort. + 6.00 ng/PUF	002	16-Jul-19	17-Jul-19	279262	155284	1.79839	0.2935184	0.0008979839	6.12397	6.00	102
113	PUF Control	003	17-Jul-19	18-Jul-19	0	129348	0.00000	0.2980736	-0.00008291312	0.00000	NA	NA
114	PUF Control	003	17-Jul-19	18-Jul-19	0	134640	0.00000	0.2980736	-0.00008291312	0.00000	NA	NA
115	PUF Fort. + 0.100 ng/PUF	003	17-Jul-19	18-Jul-19	4416	124169	0.03557	0.2980736	-0.00008291312	0.11960	0.100	120
116	PUF Fort. + 0.100 ng/PUF	003	17-Jul-19	18-Jul-19	4495	135562	0.03316	0.2980736	-0.00008291312	0.11152	0.100	112
117	PUF Fort. + 0.100 ng/PUF	003	17-Jul-19	18-Jul-19	4135	125283	0.03301	0.2980736	-0.00008291312	0.11101	0.100	111
118	PUF Fort. + 6.00 ng/PUF	003	17-Jul-19	18-Jul-19	237483	127536	1.86209	0.2980736	-0.00008291312	6.24735	6.00	104
119	PUF Fort. + 6.00 ng/PUF	003	17-Jul-19	18-Jul-19	234137	135033	1.73393	0.2980736	-0.00008291312	5.81739	6.00	97
120	PUF Fort. + 6.00 ng/PUF	003	17-Jul-19	18-Jul-19	229578	135697	1.69184	0.2980736	-0.00008291312	5.67620	6.00	95
169	PUF Control	004	18-Jul-19	18-Jul-19	0	159790	0.00000	0.3022826	-0.000281	0.00000	NA	NA

**Recovery Data Table for PUF Samples Fortified with Dicamba**

EEAL ID (89311-)	Sample Description	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)	Fort. Level (ng/PUF)	% Rec.
								m	b			
170	PUF Control	004	18-Jul-19	18-Jul-19	0	163754	0.00000	0.3022826	-0.000281	0.00000	NA	NA
171	PUF Fort. + 0.100 ng/PUF	004	18-Jul-19	18-Jul-19	4130	136088	0.03035	0.3022826	-0.000281	0.10132	0.100	101
172	PUF Fort. + 0.100 ng/PUF	004	18-Jul-19	18-Jul-19	4898	167011	0.02933	0.3022826	-0.000281	0.09795	0.100	98
173	PUF Fort. + 0.100 ng/PUF	004	18-Jul-19	18-Jul-19	6283	163769	0.03837	0.3022826	-0.000281	0.12785	0.100	128
174	PUF Fort. + 6.00 ng/PUF	004	18-Jul-19	18-Jul-19	266431	154877	1.72027	0.3022826	-0.000281	5.69187	6.00	95
175	PUF Fort. + 6.00 ng/PUF	004	18-Jul-19	18-Jul-19	287839	158446	1.81664	0.3022826	-0.000281	6.01066	6.00	100
176	PUF Fort. + 6.00 ng/PUF	004	18-Jul-19	18-Jul-19	288802	159509	1.81057	0.3022826	-0.000281	5.99060	6.00	100
225	PUF Control	005	19-Jul-19	20-Jul-19	0	180854	0.00000	0.3069103	0.002515019	0.00000	NA	NA
226	PUF Control	005	19-Jul-19	20-Jul-19	0	170551	0.00000	0.3069103	0.002515019	0.00000	NA	NA
227	PUF Fort. + 0.100 ng/PUF	005	19-Jul-19	20-Jul-19	5898	177788	0.03318	0.3069103	0.002515019	0.09990	0.100	100
228	PUF Fort. + 0.100 ng/PUF	005	19-Jul-19	20-Jul-19	5937	181619	0.03269	0.3069103	0.002515019	0.09831	0.100	98
229	PUF Fort. + 0.100 ng/PUF	005	19-Jul-19	20-Jul-19	5592	167981	0.03329	0.3069103	0.002515019	0.10028	0.100	100
230	PUF Fort. + 6.00 ng/PUF	005	19-Jul-19	20-Jul-19	316624	182135	1.73841	0.3069103	0.002515019	5.65603	6.00	94
231	PUF Fort. + 6.00 ng/PUF	005	19-Jul-19	20-Jul-19	325549	177625	1.83279	0.3069103	0.002515019	5.96354	6.00	99
232	PUF Fort. + 6.00 ng/PUF	005	19-Jul-19	20-Jul-19	325747	184086	1.76954	0.3069103	0.002515019	5.75746	6.00	96
233	PUF Fort. + 60.0 ng/PUF	005	19-Jul-19	20-Jul-19	325712	18464	17.63993	0.3069103	0.002515019	57.46766	60.0	96
234	PUF Fort. + 60.0 ng/PUF	005	19-Jul-19	20-Jul-19	330995	18405	17.98355	0.3069103	0.002515019	58.58727	60.0	98
235	PUF Fort. + 60.0 ng/PUF	005	19-Jul-19	20-Jul-19	336381	18029	18.65731	0.3069103	0.002515019	60.78255	60.0	101

**Recovery Statistics for PUF Samples Fortified with Dicamba**

	<b>0.100 ng/PUF</b>	<b>6.00 ng/PUF</b>	<b>60.0 ng/PUF</b>
<b>Number of Fortifications</b>	15	15	3
<b>Minimum Recovery (%)</b>	78	94	96
<b>Maximum Recovery (%)</b>	128	105	101
<b>Mean (%)</b>	103	99	98
<b>Standard Deviation (%)</b>	12.5	3.8	2.5
<b>Relative Standard Deviation (%)</b>	12.1	3.9	2.6

## G.2 Raw Data Table for PUF Sample Analysis

The *Raw Data Table for PUF Sample Analysis* contains data from which result summary Table 6-Table 9 were generated. The description of each column is presented below.

### A. EEAL ID

The analytical ID used to track the sample through analysis.

### B. Sample ID

The field ID which identifies trial, sample type, location or distance, timing, treatment, and replicate for each sample analyzed.

### C. Set No.

Analytical set number

### D. Extraction Date

The date the samples were extracted in the analytical laboratory.

### E. Analysis Date

The date the analytical results were acquired from the instrument.

### F. Peak Area

Peak area of the analyte

### G. IS Peak Area

Peak area of the internal standard

### H. Area Ratio

The ratio of the area of the analyte to the area of the internal standard determined according to the equation:

$$Area\ Ratio = \frac{Peak\ Area}{IS\ Peak\ Area}$$

### I. Curve Constants (m and b)

Calculations for instrumental analysis were conducted using a validated software application (e.g., Analyst<sup>®</sup> 1.6.3 Software) to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. A weighted linear curve (1/x) is used. The resulting equation defining the standard curve is shown below:

$$y = mx + b$$

where:

y = area ratio of the analyte  
m = slope of curve  
x = detector response in ng/PUF  
b = y-intercept of curve

The calculation performed by the instrument may be checked manually by applying the following equation:

$$x \text{ (ng/PUF)} = \frac{y - b}{m}$$

The curve constants (slope and intercept) were calculated by the computer systems to a higher number of significant digits than is reported in the raw data and recovery data tables. The amounts of dicamba found presented here were calculated based on the higher number of significant digits; therefore, hand calculations based on these values may contain small rounding differences.

#### **J. Dicamba (ng/PUF)**

Dicamba residue found in ng of analyte per PUF sample collector obtained from Analyst<sup>®</sup>.

#### **K. Example Calculation – Treated Sample**

From the Raw Data Table:

EEAL ID: 89311-009  
Sample ID: REG-2019-0035 . PCM . 015 . PRE . 1 . 001  
Analytical Set: 001

where:

peak area = 3722  
IS peak area = 155393  
area ratio (y) = 0.02395  
y-intercept (b) = 0.00138691  
slope (m) = 0.2965074

$$\text{Area Ratio} = \frac{3722}{155393} = 0.02395$$

$$\text{Dicamba (ng/PUF)} = \frac{(0.02395 - 0.00138691)}{0.2965074} = 0.07610 \text{ ng/PUF}$$

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
009	REG-2019-0035 . PCM . 015 . PRE . 1 . 001	001	15-Jul-19	16-Jul-19	3722	155393	0.02395	0.2965074	0.00138691	0.07610
010	REG-2019-0035 . PCM . 015 . PRE . 2 . 002	001	15-Jul-19	16-Jul-19	3840	156921	0.02447	0.2965074	0.00138691	0.07784
011	REG-2019-0035 . PCM . 015 . 0-6 hr . 1 . 025	001	15-Jul-19	16-Jul-19	379465	153405	2.47361	0.2965074	0.00138691	8.33782
012	REG-2019-0035 . PCM . 033 . 0-6 hr . 1 . 026	001	15-Jul-19	16-Jul-19	342632	169369	2.02299	0.2965074	0.00138691	6.81806
013	REG-2019-0035 . PCM . 055 . 0-6 hr . 1 . 027	001	15-Jul-19	16-Jul-19	147168	106974	1.37573	0.2965074	0.00138691	4.63512
014	REG-2019-0035 . PCM . 090 . 0-6 hr . 1 . 028	001	15-Jul-19	16-Jul-19	136487	137171	0.99501	0.2965074	0.00138691	3.35110
015	REG-2019-0035 . PCM . 150 . 0-6 hr . 1 . 029	001	15-Jul-19	16-Jul-19	116838	129333	0.90339	0.2965074	0.00138691	3.04209
016	REG-2019-0035 . PCM . 150 . 0-6 hr . 2 . 030	001	15-Jul-19	16-Jul-19	121578	159377	0.76284	0.2965074	0.00138691	2.56806
025	REG-2019-0035 . PCM . 015 . 6-12 . 1 . 039	001	15-Jul-19	16-Jul-19	38000	140462	0.27053	0.2965074	0.00138691	0.90772
026	REG-2019-0035 . PCM . 033 . 6-12 . 1 . 040	001	15-Jul-19	16-Jul-19	36699	133159	0.27560	0.2965074	0.00138691	0.92482
027	REG-2019-0035 . PCM . 055 . 6-12 . 1 . 041	001	15-Jul-19	16-Jul-19	18380	111986	0.16413	0.2965074	0.00138691	0.54885
028	REG-2019-0035 . PCM . 090 . 6-12 . 1 . 042	001	15-Jul-19	16-Jul-19	25029	146045	0.17138	0.2965074	0.00138691	0.57331
029	REG-2019-0035 . PCM . 150 . 6-12 . 1 . 043	001	15-Jul-19	16-Jul-19	17089	143325	0.11923	0.2965074	0.00138691	0.39745
030	REG-2019-0035 . PCM . 150 . 6-12 . 2 . 044	001	15-Jul-19	16-Jul-19	16490	132066	0.12487	0.2965074	0.00138691	0.41644
039	REG-2019-0035 . PCM . 015 . 12-24 hr . 1 . 053	001	15-Jul-19	16-Jul-19	95464	140003	0.68187	0.2965074	0.00138691	2.29501
040	REG-2019-0035 . PCM . 033 . 12-24 hr . 1 . 054	001	15-Jul-19	16-Jul-19	65651	104219	0.62993	0.2965074	0.00138691	2.11983
041	REG-2019-0035 . PCM . 055 . 12-24 hr . 1 . 055	001	15-Jul-19	16-Jul-19	113850	145048	0.78491	0.2965074	0.00138691	2.64252
042	REG-2019-0035 . PCM . 090 . 12-24 hr . 1 . 056	001	15-Jul-19	17-Jul-19	0	116261	0.00000	0.2965074	0.00138691	0.00000
043	REG-2019-0035 . PCM . 150 . 12-24 hr . 1 . 057	001	15-Jul-19	17-Jul-19	42172	132073	0.31931	0.2965074	0.00138691	1.07221
044	REG-2019-0035 . PCM . 150 . 12-24 hr . 2 . 058	001	15-Jul-19	17-Jul-19	50179	140380	0.35745	0.2965074	0.00138691	1.20087
065	REG-2019-0035 . PCM . 015 . 24-36 hr . 1 . 067	002	16-Jul-19	17-Jul-19	64613	116315	0.55550	0.2935184	0.0008979839	1.88951
066	REG-2019-0035 . PCM . 033 . 24-36 hr . 1 . 068	002	16-Jul-19	17-Jul-19	42683	112832	0.37828	0.2935184	0.0008979839	1.28573
067	REG-2019-0035 . PCM . 055 . 24-36 hr . 1 . 069	002	16-Jul-19	17-Jul-19	37827	105786	0.35758	0.2935184	0.0008979839	1.21520
068	REG-2019-0035 . PCM . 090 . 24-36 hr . 1 . 070	002	16-Jul-19	17-Jul-19	25560	91148	0.28042	0.2935184	0.0008979839	0.95233
069	REG-2019-0035 . PCM . 150 . 24-36 hr . 1 . 071	002	16-Jul-19	17-Jul-19	26600	88905	0.29920	0.2935184	0.0008979839	1.01630



### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
070	REG-2019-0035 . PCM . 150 . 24-36 hr . 2 . 072	002	16-Jul-19	17-Jul-19	28173	115744	0.24341	0.2935184	0.0008979839	0.82621
075	REG-2019-0035 . PCM . 015 . 36-48 hr . 1 . 081	002	16-Jul-19	17-Jul-19	166136	107871	1.54012	0.2935184	0.0008979839	5.24406
076	REG-2019-0035 . PCM . 033 . 36-48 hr . 1 . 082	002	16-Jul-19	17-Jul-19	61607	103727	0.59394	0.2935184	0.0008979839	2.02044
077	REG-2019-0035 . PCM . 055 . 36-48 hr . 1 . 083	002	16-Jul-19	17-Jul-19	34805	117053	0.29734	0.2935184	0.0008979839	1.00996
078	REG-2019-0035 . PCM . 090 . 36-48 hr . 1 . 084	002	16-Jul-19	17-Jul-19	17871	105408	0.16954	0.2935184	0.0008979839	0.57455
079	REG-2019-0035 . PCM . 150 . 36-48 hr . 1 . 085	002	16-Jul-19	17-Jul-19	13929	100668	0.13837	0.2935184	0.0008979839	0.46836
080	REG-2019-0035 . PCM . 150 . 36-48 hr . 2 . 086	002	16-Jul-19	17-Jul-19	14642	101771	0.14387	0.2935184	0.0008979839	0.48709
089	REG-2019-0035 . PCM . 015 . 48-60 hr . 1 . 095	002	16-Jul-19	17-Jul-19	19158	114257	0.16768	0.2935184	0.0008979839	0.56820
090	REG-2019-0035 . PCM . 033 . 48-60 hr . 1 . 096	002	16-Jul-19	17-Jul-19	18372	109726	0.16743	0.2935184	0.0008979839	0.56738
091	REG-2019-0035 . PCM . 055 . 48-60 hr . 1 . 097	002	16-Jul-19	17-Jul-19	14048	90477	0.15526	0.2935184	0.0008979839	0.52590
092	REG-2019-0035 . PCM . 090 . 48-60 hr . 1 . 098	002	16-Jul-19	17-Jul-19	15493	103880	0.14914	0.2935184	0.0008979839	0.50506
093	REG-2019-0035 . PCM . 150 . 48-60 hr . 1 . 099	002	16-Jul-19	17-Jul-19	15535	111430	0.13941	0.2935184	0.0008979839	0.47190
094	REG-2019-0035 . PCM . 150 . 48-60 hr . 2 . 100	002	16-Jul-19	17-Jul-19	17589	112060	0.15696	0.2935184	0.0008979839	0.53170
103	REG-2019-0035 . PCM . 015 . 60-72 hr . 1 . 109	002	16-Jul-19	17-Jul-19	17318	109075	0.15877	0.2935184	0.0008979839	0.53787
104	REG-2019-0035 . PCM . 033 . 60-72 hr . 1 . 110	002	16-Jul-19	17-Jul-19	16148	106102	0.15219	0.2935184	0.0008979839	0.51545
105	REG-2019-0035 . PCM . 055 . 60-72 hr . 1 . 111	002	16-Jul-19	18-Jul-19	16477	110372	0.14929	0.2935184	0.0008979839	0.50555
106	REG-2019-0035 . PCM . 090 . 60-72 hr . 1 . 112	002	16-Jul-19	18-Jul-19	15837	107150	0.14780	0.2935184	0.0008979839	0.50050
107	REG-2019-0035 . PCM . 150 . 60-72 hr . 1 . 113	002	16-Jul-19	18-Jul-19	10166	104189	0.09757	0.2935184	0.0008979839	0.32936
108	REG-2019-0035 . PCM . 150 . 60-72 hr . 2 . 114	002	16-Jul-19	18-Jul-19	13523	105834	0.12777	0.2935184	0.0008979839	0.43226
125	REG-2019-0035 . PCM . 015 . 72-84 hr . 1 . 123	003	17-Jul-19	18-Jul-19	12202	118657	0.10283	0.2980736	-0.00008291312	0.34527
126	REG-2019-0035 . PCM . 033 . 72-84 hr . 1 . 124	003	17-Jul-19	18-Jul-19	10701	120452	0.08884	0.2980736	-0.00008291312	0.29832
127	REG-2019-0035 . PCM . 055 . 72-84 hr . 1 . 125	003	17-Jul-19	18-Jul-19	8029	101782	0.07889	0.2980736	-0.00008291312	0.26493
128	REG-2019-0035 . PCM . 090 . 72-84 hr . 1 . 126	003	17-Jul-19	18-Jul-19	10056	125491	0.08013	0.2980736	-0.00008291312	0.26911
129	REG-2019-0035 . PCM . 150 . 72-84 hr . 1 . 127	003	17-Jul-19	18-Jul-19	9285	118811	0.07815	0.2980736	-0.00008291312	0.26246
130	REG-2019-0035 . PCM . 150 . 72-84 hr . 2 . 128	003	17-Jul-19	18-Jul-19	6943	100502	0.06908	0.2980736	-0.00008291312	0.23203

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
139	REG-2019-0035 . PCM . 015 . 84-96 hr . 1 . 137	003	17-Jul-19	18-Jul-19	39262	130295	0.30133	0.2980736	-0.00008291312	1.01120
140	REG-2019-0035 . PCM . 033 . 84-96 hr . 1 . 138	003	17-Jul-19	18-Jul-19	51523	118064	0.43640	0.2980736	-0.00008291312	1.46435
141	REG-2019-0035 . PCM . 055 . 84-96 hr . 1 . 139	003	17-Jul-19	18-Jul-19	8258	118133	0.06990	0.2980736	-0.00008291312	0.23479
142	REG-2019-0035 . PCM . 090 . 84-96 hr . 1 . 140	003	17-Jul-19	18-Jul-19	6141	105279	0.05833	0.2980736	-0.00008291312	0.19597
143	REG-2019-0035 . PCM . 150 . 84-96 hr . 1 . 141	003	17-Jul-19	18-Jul-19	4301	125413	0.03429	0.2980736	-0.00008291312	0.11532
144	REG-2019-0035 . PCM . 150 . 84-96 hr . 2 . 142	003	17-Jul-19	18-Jul-19	4582	116978	0.03917	0.2980736	-0.00008291312	0.13169
153	REG-2019-0035 . PCM . 015 . 96-108 hr . 1 . 151	003	17-Jul-19	18-Jul-19	26797	101624	0.26369	0.2980736	-0.00008291312	0.88493
154	REG-2019-0035 . PCM . 033 . 96-108 hr . 1 . 152	003	17-Jul-19	18-Jul-19	20764	109959	0.18883	0.2980736	-0.00008291312	0.63380
155	REG-2019-0035 . PCM . 055 . 96-108 hr . 1 . 153	003	17-Jul-19	18-Jul-19	7277	119479	0.06090	0.2980736	-0.00008291312	0.20461
156	REG-2019-0035 . PCM . 090 . 96-108 hr . 1 . 154	003	17-Jul-19	18-Jul-19	5165	112430	0.04594	0.2980736	-0.00008291312	0.15439
157	REG-2019-0035 . PCM . 150 . 96-108 hr . 1 . 155	003	17-Jul-19	18-Jul-19	8544	99689	0.08571	0.2980736	-0.00008291312	0.28782
158	REG-2019-0035 . PCM . 150 . 96-108 hr . 2 . 156	003	17-Jul-19	18-Jul-19	6420	106088	0.06052	0.2980736	-0.00008291312	0.20330
159	REG-2019-0035 . PCM . 015 . 108-120 hr . 1 . 165	003	17-Jul-19	18-Jul-19	11394	119867	0.09505	0.2980736	-0.00008291312	0.31916
160	REG-2019-0035 . PCM . 033 . 108-120 hr . 1 . 166	003	17-Jul-19	18-Jul-19	8843	112381	0.07869	0.2980736	-0.00008291312	0.26426
161	REG-2019-0035 . PCM . 055 . 108-120 hr . 1 . 167	003	17-Jul-19	18-Jul-19	11192	99499	0.11249	0.2980736	-0.00008291312	0.37766
162	REG-2019-0035 . PCM . 090 . 108-120 hr . 1 . 168	003	17-Jul-19	18-Jul-19	10343	117440	0.08807	0.2980736	-0.00008291312	0.29574
163	REG-2019-0035 . PCM . 150 . 108-120 hr . 1 . 169	003	17-Jul-19	18-Jul-19	11127	112825	0.09862	0.2980736	-0.00008291312	0.33113
164	REG-2019-0035 . PCM . 150 . 108-120 hr . 2 . 170	003	17-Jul-19	18-Jul-19	8872	110846	0.08004	0.2980736	-0.00008291312	0.26881
189	REG-2019-0035 . PCM . 015 . 120-132 hr . 1 . 179	004	18-Jul-19	18-Jul-19	15228	150167	0.10141	0.3022826	-0.000281	0.33640
190	REG-2019-0035 . PCM . 033 . 120-132 hr . 1 . 180	004	18-Jul-19	18-Jul-19	17323	131232	0.13200	0.3022826	-0.000281	0.43761
191	REG-2019-0035 . PCM . 055 . 120-132 hr . 1 . 181	004	18-Jul-19	18-Jul-19	21884	133686	0.16370	0.3022826	-0.000281	0.54247
192	REG-2019-0035 . PCM . 090 . 120-132 hr . 1 . 182	004	18-Jul-19	18-Jul-19	12041	141346	0.08519	0.3022826	-0.000281	0.28274
193	REG-2019-0035 . PCM . 150 . 120-132 hr . 1 . 183	004	18-Jul-19	18-Jul-19	17018	145246	0.11717	0.3022826	-0.000281	0.38854
194	REG-2019-0035 . PCM . 150 . 120-132 hr . 2 . 184	004	18-Jul-19	18-Jul-19	12063	118023	0.10221	0.3022826	-0.000281	0.33904
203	REG-2019-0035 . PCM . 015 . 132-144 hr . 1 . 193	004	18-Jul-19	18-Jul-19	17192	107838	0.15943	0.3022826	-0.000281	0.52834

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
204	REG-2019-0035 . PCM . 033 . 132-144 hr . 1 . 194	004	18-Jul-19	18-Jul-19	18637	108099	0.17241	0.3022826	-0.000281	0.57128
205	REG-2019-0035 . PCM . 055 . 132-144 hr . 1 . 195	004	18-Jul-19	18-Jul-19	22631	131024	0.17272	0.3022826	-0.000281	0.57232
206	REG-2019-0035 . PCM . 090 . 132-144 hr . 1 . 196	004	18-Jul-19	18-Jul-19	22006	121793	0.18068	0.3022826	-0.000281	0.59866
207	REG-2019-0035 . PCM . 150 . 132-144 hr . 1 . 197	004	18-Jul-19	18-Jul-19	21914	125243	0.17497	0.3022826	-0.000281	0.57976
208	REG-2019-0035 . PCM . 150 . 132-144 hr . 2 . 198	004	18-Jul-19	18-Jul-19	23817	121658	0.19577	0.3022826	-0.000281	0.64856
236	REG-2019-0035 . PCM . 015 . 144-156 hr . 1 . 207	005	19-Jul-19	20-Jul-19	15037	156273	0.09622	0.3069103	0.002515019	0.30532
237	REG-2019-0035 . PCM . 033 . 144-156 hr . 1 . 208	005	19-Jul-19	20-Jul-19	136811	153805	0.88951	0.3069103	0.002515019	2.89009
238	REG-2019-0035 . PCM . 055 . 144-156 hr . 1 . 209	005	19-Jul-19	20-Jul-19	17923	144316	0.12419	0.3069103	0.002515019	0.39646
239	REG-2019-0035 . PCM . 090 . 144-156 hr . 1 . 210	005	19-Jul-19	20-Jul-19	15723	161472	0.09737	0.3069103	0.002515019	0.30908
240	REG-2019-0035 . PCM . 150 . 144-156 hr . 1 . 211	005	19-Jul-19	20-Jul-19	34798	148215	0.23478	0.3069103	0.002515019	0.75679
241	REG-2019-0035 . PCM . 150 . 144-156 hr . 2 . 212	005	19-Jul-19	20-Jul-19	17446	146350	0.11921	0.3069103	0.002515019	0.38022
242	REG-2019-0035 . PCM . 015 . 156-168 hr . 1 . 221	005	19-Jul-19	20-Jul-19	38245	148130	0.25818	0.3069103	0.002515019	0.83304
243	REG-2019-0035 . PCM . 033 . 156-168 hr . 1 . 222	005	19-Jul-19	20-Jul-19	43183	151393	0.28524	0.3069103	0.002515019	0.92119
244	REG-2019-0035 . PCM . 055 . 156-168 hr . 1 . 223	005	19-Jul-19	20-Jul-19	44223	139387	0.31727	0.3069103	0.002515019	1.02555
245	REG-2019-0035 . PCM . 090 . 156-168 hr . 1 . 224	005	19-Jul-19	20-Jul-19	56516	179715	0.31448	0.3069103	0.002515019	1.01645
246	REG-2019-0035 . PCM . 150 . 156-168 hr . 1 . 225	005	19-Jul-19	20-Jul-19	74990	171233	0.43794	0.3069103	0.002515019	1.41873
247	REG-2019-0035 . PCM . 150 . 156-168 hr . 2 . 226	005	19-Jul-19	20-Jul-19	57800	167838	0.34438	0.3069103	0.002515019	1.11389
017	REG-2019-0035 . POF . A . 0-6 hr . 1 . 031	001	15-Jul-19	16-Jul-19	81907	129448	0.63274	0.2965074	0.00138691	2.12931
018	REG-2019-0035 . POF . B . 0-6 hr . 1 . 032	001	15-Jul-19	16-Jul-19	54551	150071	0.36350	0.2965074	0.00138691	1.22126
019	REG-2019-0035 . POF . C . 0-6 hr . 1 . 033	001	15-Jul-19	16-Jul-19	107451	140907	0.76256	0.2965074	0.00138691	2.56714
020	REG-2019-0035 . POF . D . 0-6 hr . 1 . 034	001	15-Jul-19	16-Jul-19	68533	151865	0.45128	0.2965074	0.00138691	1.51730
021	REG-2019-0035 . POF . E . 0-6 hr . 1 . 035	001	15-Jul-19	16-Jul-19	42980	126371	0.34011	0.2965074	0.00138691	1.14237
022	REG-2019-0035 . POF . F . 0-6 hr . 1 . 036	001	15-Jul-19	16-Jul-19	4600	108430	0.04242	0.2965074	0.00138691	0.13839
023	REG-2019-0035 . POF . G . 0-6 hr . 1 . 037	001	15-Jul-19	16-Jul-19	6718	128038	0.05247	0.2965074	0.00138691	0.17229
024	REG-2019-0035 . POF . H . 0-6 hr . 1 . 038	001	15-Jul-19	16-Jul-19	14397	150716	0.09553	0.2965074	0.00138691	0.31749

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
031	REG-2019-0035 . POF . A . 6-12 hr . 1 . 045	001	15-Jul-19	16-Jul-19	16884	151609	0.11136	0.2965074	0.00138691	0.37090
032	REG-2019-0035 . POF . B . 6-12 hr . 1 . 046	001	15-Jul-19	16-Jul-19	13811	158112	0.08735	0.2965074	0.00138691	0.28991
033	REG-2019-0035 . POF . C . 6-12 hr . 1 . 047	001	15-Jul-19	16-Jul-19	15539	152690	0.10177	0.2965074	0.00138691	0.33854
034	REG-2019-0035 . POF . D . 6-12 hr . 1 . 048	001	15-Jul-19	16-Jul-19	7691	149781	0.05135	0.2965074	0.00138691	0.16851
035	REG-2019-0035 . POF . E . 6-12 hr . 1 . 049	001	15-Jul-19	16-Jul-19	8156	162452	0.05021	0.2965074	0.00138691	0.16465
036	REG-2019-0035 . POF . F . 6-12 hr . 1 . 050	001	15-Jul-19	16-Jul-19	12144	151979	0.07991	0.2965074	0.00138691	0.26481
037	REG-2019-0035 . POF . G . 6-12 hr . 1 . 051	001	15-Jul-19	16-Jul-19	9407	137645	0.06834	0.2965074	0.00138691	0.22582
038	REG-2019-0035 . POF . H . 6-12 hr . 1 . 052	001	15-Jul-19	16-Jul-19	5718	153216	0.03732	0.2965074	0.00138691	0.12119
045	REG-2019-0035 . POF . A . 12-24 hr . 1 . 059	001	15-Jul-19	17-Jul-19	69951	149240	0.46872	0.2965074	0.00138691	1.57612
046	REG-2019-0035 . POF . B . 12-24 hr . 1 . 060	001	15-Jul-19	17-Jul-19	32810	100503	0.32646	0.2965074	0.00138691	1.09634
047	REG-2019-0035 . POF . C . 12-24 hr . 1 . 061	001	15-Jul-19	17-Jul-19	145761	149221	0.97682	0.2965074	0.00138691	3.28974
048	REG-2019-0035 . POF . D . 12-24 hr . 1 . 062	001	15-Jul-19	17-Jul-19	24814	132707	0.18698	0.2965074	0.00138691	0.62594
049	REG-2019-0035 . POF . E . 12-24 hr . 1 . 063	001	15-Jul-19	17-Jul-19	29760	116906	0.25456	0.2965074	0.00138691	0.85386
050	REG-2019-0035 . POF . F . 12-24 hr . 1 . 064	001	15-Jul-19	17-Jul-19	24972	110912	0.22515	0.2965074	0.00138691	0.75466
051	REG-2019-0035 . POF . G . 12-24 hr . 1 . 065	001	15-Jul-19	17-Jul-19	32680	144372	0.22636	0.2965074	0.00138691	0.75875
052	REG-2019-0035 . POF . H . 12-24 hr . 1 . 066	001	15-Jul-19	17-Jul-19	31409	135075	0.23253	0.2965074	0.00138691	0.77956
053	REG-2019-0035 . POF . A . 24-36 hr . 1 . 073	001	15-Jul-19	17-Jul-19	17585	148047	0.11878	0.2965074	0.00138691	0.39592
054	REG-2019-0035 . POF . B . 24-36 hr . 1 . 074	001	15-Jul-19	17-Jul-19	18815	134626	0.13976	0.2965074	0.00138691	0.46666
055	REG-2019-0035 . POF . C . 24-36 hr . 1 . 075	001	15-Jul-19	17-Jul-19	68162	137970	0.49403	0.2965074	0.00138691	1.66149
056	REG-2019-0035 . POF . D . 24-36 hr . 1 . 076	001	15-Jul-19	17-Jul-19	13306	127732	0.10417	0.2965074	0.00138691	0.34664
071	REG-2019-0035 . POF . E . 24-36 hr . 1 . 077	002	16-Jul-19	17-Jul-19	22466	122291	0.18371	0.2935184	0.0008979839	0.62283
072	REG-2019-0035 . POF . F . 24-36 hr . 1 . 078	002	16-Jul-19	17-Jul-19	7170	130235	0.05505	0.2935184	0.0008979839	0.18450
073	REG-2019-0035 . POF . G . 24-36 hr . 1 . 079	002	16-Jul-19	17-Jul-19	6241	124917	0.04996	0.2935184	0.0008979839	0.16716
074	REG-2019-0035 . POF . H . 24-36 hr . 1 . 080	002	16-Jul-19	17-Jul-19	5811	102863	0.05650	0.2935184	0.0008979839	0.18942
081	REG-2019-0035 . POF . A . 36-48 hr . 1 . 087	002	16-Jul-19	17-Jul-19	9683	108930	0.08889	0.2935184	0.0008979839	0.29980

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
082	REG-2019-0035 . POF . B . 36-48 hr . 1 . 088	002	16-Jul-19	17-Jul-19	11776	111982	0.10516	0.2935184	0.0008979839	0.35522
083	REG-2019-0035 . POF . C . 36-48 hr . 1 . 089	002	16-Jul-19	17-Jul-19	339874	138957	2.44589	0.2935184	0.0008979839	8.32994
084	REG-2019-0035 . POF . D . 36-48 hr . 1 . 090	002	16-Jul-19	17-Jul-19	7036	119009	0.05912	0.2935184	0.0008979839	0.19838
085	REG-2019-0035 . POF . E . 36-48 hr . 1 . 091	002	16-Jul-19	17-Jul-19	11072	120035	0.09224	0.2935184	0.0008979839	0.31120
086	REG-2019-0035 . POF . F . 36-48 hr . 1 . 092	002	16-Jul-19	17-Jul-19	10662	105599	0.10097	0.2935184	0.0008979839	0.34093
087	REG-2019-0035 . POF . G . 36-48 hr . 1 . 093	002	16-Jul-19	17-Jul-19	32810	113432	0.28925	0.2935184	0.0008979839	0.98239
088	REG-2019-0035 . POF . H . 36-48 hr . 1 . 094	002	16-Jul-19	17-Jul-19	13502	109234	0.12360	0.2935184	0.0008979839	0.41805
095	REG-2019-0035 . POF . A . 48-60 hr . 1 . 101	002	16-Jul-19	17-Jul-19	13588	88517	0.15351	0.2935184	0.0008979839	0.51994
096	REG-2019-0035 . POF . B . 48-60 hr . 1 . 102	002	16-Jul-19	17-Jul-19	13115	106090	0.12362	0.2935184	0.0008979839	0.41809
097	REG-2019-0035 . POF . C . 48-60 hr . 1 . 103	002	16-Jul-19	17-Jul-19	62992	101489	0.62068	0.2935184	0.0008979839	2.11157
098	REG-2019-0035 . POF . D . 48-60 hr . 1 . 104	002	16-Jul-19	17-Jul-19	12453	103521	0.12030	0.2935184	0.0008979839	0.40678
099	REG-2019-0035 . POF . E . 48-60 hr . 1 . 105	002	16-Jul-19	17-Jul-19	15934	103191	0.15442	0.2935184	0.0008979839	0.52303
100	REG-2019-0035 . POF . F . 48-60 hr . 1 . 106	002	16-Jul-19	17-Jul-19	15612	107822	0.14479	0.2935184	0.0008979839	0.49023
101	REG-2019-0035 . POF . G . 48-60 hr . 1 . 107	002	16-Jul-19	17-Jul-19	16466	107609	0.15301	0.2935184	0.0008979839	0.51825
102	REG-2019-0035 . POF . H . 48-60 hr . 1 . 108	002	16-Jul-19	17-Jul-19	14980	114080	0.13131	0.2935184	0.0008979839	0.44431
109	REG-2019-0035 . POF . A . 60-72 hr . 1 . 115	002	16-Jul-19	18-Jul-19	11816	104827	0.11272	0.2935184	0.0008979839	0.38097
110	REG-2019-0035 . POF . B . 60-72 hr . 1 . 116	002	16-Jul-19	18-Jul-19	11747	105932	0.11089	0.2935184	0.0008979839	0.37474
111	REG-2019-0035 . POF . C . 60-72 hr . 1 . 117	002	16-Jul-19	18-Jul-19	14682	111039	0.13223	0.2935184	0.0008979839	0.44743
112	REG-2019-0035 . POF . D . 60-72 hr . 1 . 118	002	16-Jul-19	18-Jul-19	13294	97800	0.13593	0.2935184	0.0008979839	0.46004
121	REG-2019-0035 . POF . E . 60-72 hr . 1 . 119	003	17-Jul-19	18-Jul-19	21908	131951	0.16603	0.2980736	-0.00008291312	0.55730
122	REG-2019-0035 . POF . F . 60-72 hr . 1 . 120	003	17-Jul-19	18-Jul-19	20725	128591	0.16117	0.2980736	-0.00008291312	0.54098
123	REG-2019-0035 . POF . G . 60-72 hr . 1 . 121	003	17-Jul-19	18-Jul-19	52587	132991	0.39541	0.2980736	-0.00008291312	1.32684
124	REG-2019-0035 . POF . H . 60-72 hr . 1 . 122	003	17-Jul-19	18-Jul-19	17386	121185	0.14346	0.2980736	-0.00008291312	0.48158
131	REG-2019-0035 . POF . A . 72-84 hr . 1 . 129	003	17-Jul-19	18-Jul-19	8394	107438	0.07812	0.2980736	-0.00008291312	0.26238
132	REG-2019-0035 . POF . B . 72-84 hr . 1 . 130	003	17-Jul-19	18-Jul-19	9698	123281	0.07867	0.2980736	-0.00008291312	0.26420

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
133	REG-2019-0035 . POF . C . 72-84 hr . 1 . 131	003	17-Jul-19	18-Jul-19	9377	95437	0.09826	0.2980736	-0.00008291312	0.32991
134	REG-2019-0035 . POF . D . 72-84 hr . 1 . 132	003	17-Jul-19	18-Jul-19	6620	105705	0.06262	0.2980736	-0.00008291312	0.21037
135	REG-2019-0035 . POF . E . 72-84 hr . 1 . 133	003	17-Jul-19	18-Jul-19	9849	132408	0.07438	0.2980736	-0.00008291312	0.24982
136	REG-2019-0035 . POF . F . 72-84 hr . 1 . 134	003	17-Jul-19	18-Jul-19	5694	96741	0.05886	0.2980736	-0.00008291312	0.19775
137	REG-2019-0035 . POF . G . 72-84 hr . 1 . 135	003	17-Jul-19	18-Jul-19	8060	126675	0.06363	0.2980736	-0.00008291312	0.21374
138	REG-2019-0035 . POF . H . 72-84 hr . 1 . 136	003	17-Jul-19	18-Jul-19	7144	105194	0.06791	0.2980736	-0.00008291312	0.22812
145	REG-2019-0035 . POF . A . 84-96 hr . 1 . 143	003	17-Jul-19	18-Jul-19	3137	124133	0.02527	0.2980736	-0.00008291312	0.08507
146	REG-2019-0035 . POF . B . 84-96 hr . 1 . 144	003	17-Jul-19	18-Jul-19	4118	111956	0.03678	0.2980736	-0.00008291312	0.12369
147	REG-2019-0035 . POF . C . 84-96 hr . 1 . 145	003	17-Jul-19	18-Jul-19	5554	112025	0.04957	0.2980736	-0.00008291312	0.16659
148	REG-2019-0035 . POF . D . 84-96 hr . 1 . 146	003	17-Jul-19	18-Jul-19	8746	127065	0.06883	0.2980736	-0.00008291312	0.23120
149	REG-2019-0035 . POF . E . 84-96 hr . 1 . 147	003	17-Jul-19	18-Jul-19	3566	94537	0.03772	0.2980736	-0.00008291312	0.12682
150	REG-2019-0035 . POF . F . 84-96 hr . 1 . 148	003	17-Jul-19	18-Jul-19	4423	122340	0.03616	0.2980736	-0.00008291312	0.12157
151	REG-2019-0035 . POF . G . 84-96 hr . 1 . 149	003	17-Jul-19	18-Jul-19	5490	122196	0.04493	0.2980736	-0.00008291312	0.15100
152	REG-2019-0035 . POF . H . 84-96 hr . 1 . 150	003	17-Jul-19	18-Jul-19	4275	136703	0.03127	0.2980736	-0.00008291312	0.10520
165	REG-2019-0035 . POF . A . 96-108 hr . 1 . 157	003	17-Jul-19	18-Jul-19	3843	110834	0.03467	0.2980736	-0.00008291312	0.11660
166	REG-2019-0035 . POF . B . 96-108 hr . 1 . 158	003	17-Jul-19	18-Jul-19	3658	115631	0.03163	0.2980736	-0.00008291312	0.10640
167	REG-2019-0035 . POF . C . 96-108 hr . 1 . 159	003	17-Jul-19	18-Jul-19	11819	115084	0.10270	0.2980736	-0.00008291312	0.34481
168	REG-2019-0035 . POF . D . 96-108 hr . 1 . 160	003	17-Jul-19	18-Jul-19	4734	86885	0.05449	0.2980736	-0.00008291312	0.18309
177	REG-2019-0035 . POF . E . 96-108 hr . 1 . 161	004	18-Jul-19	18-Jul-19	7440	136404	0.05455	0.3022826	-0.000281	0.18138
178	REG-2019-0035 . POF . F . 96-108 hr . 1 . 162	004	18-Jul-19	18-Jul-19	4368	121865	0.03584	0.3022826	-0.000281	0.11951
179	REG-2019-0035 . POF . G . 96-108 hr . 1 . 163	004	18-Jul-19	18-Jul-19	9340	161347	0.05789	0.3022826	-0.000281	0.19243
180	REG-2019-0035 . POF . H . 96-108 hr . 1 . 164	004	18-Jul-19	18-Jul-19	6411	173088	0.03704	0.3022826	-0.000281	0.12345
181	REG-2019-0035 . POF . A . 108-120 hr . 1 . 171	004	18-Jul-19	18-Jul-19	43327	144370	0.30011	0.3022826	-0.000281	0.99375
182	REG-2019-0035 . POF . B . 108-120 hr . 1 . 172	004	18-Jul-19	18-Jul-19	11763	134407	0.08752	0.3022826	-0.000281	0.29045
183	REG-2019-0035 . POF . C . 108-120 hr . 1 . 173	004	18-Jul-19	18-Jul-19	14768	145019	0.10184	0.3022826	-0.000281	0.33783

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
184	REG-2019-0035 . POF . D . 108-120 hr . 1 . 174	004	18-Jul-19	18-Jul-19	12030	155834	0.07720	0.3022826	-0.000281	0.25631
185	REG-2019-0035 . POF . E . 108-120 hr . 1 . 175	004	18-Jul-19	18-Jul-19	10791	145321	0.07426	0.3022826	-0.000281	0.24659
186	REG-2019-0035 . POF . F . 108-120 hr . 1 . 176	004	18-Jul-19	18-Jul-19	9446	134348	0.07031	0.3022826	-0.000281	0.23353
187	REG-2019-0035 . POF . G . 108-120 hr . 1 . 177	004	18-Jul-19	18-Jul-19	11133	137596	0.08091	0.3022826	-0.000281	0.26859
188	REG-2019-0035 . POF . H . 108-120 hr . 1 . 178	004	18-Jul-19	18-Jul-19	12210	151341	0.08068	0.3022826	-0.000281	0.26784
195	REG-2019-0035 . POF . A . 120-132 hr . 1 . 185	004	18-Jul-19	18-Jul-19	17161	133486	0.12856	0.3022826	-0.000281	0.42622
196	REG-2019-0035 . POF . B . 120-132 hr . 1 . 186	004	18-Jul-19	18-Jul-19	10249	128014	0.08006	0.3022826	-0.000281	0.26578
197	REG-2019-0035 . POF . C . 120-132 hr . 1 . 187	004	18-Jul-19	18-Jul-19	21959	110739	0.19830	0.3022826	-0.000281	0.65693
198	REG-2019-0035 . POF . D . 120-132 hr . 1 . 188	004	18-Jul-19	18-Jul-19	9006	116918	0.07702	0.3022826	-0.000281	0.25574
199	REG-2019-0035 . POF . E . 120-132 hr . 1 . 189	004	18-Jul-19	18-Jul-19	10180	129950	0.07834	0.3022826	-0.000281	0.26009
200	REG-2019-0035 . POF . F . 120-132 hr . 1 . 190	004	18-Jul-19	18-Jul-19	10061	152938	0.06579	0.3022826	-0.000281	0.21856
201	REG-2019-0035 . POF . G . 120-132 hr . 1 . 191	004	18-Jul-19	18-Jul-19	7796	122855	0.06346	0.3022826	-0.000281	0.21086
202	REG-2019-0035 . POF . H . 120-132 hr . 1 . 192	004	18-Jul-19	18-Jul-19	14186	130612	0.10862	0.3022826	-0.000281	0.36025
209	REG-2019-0035 . POF . A . 132-144 hr . 1 . 199	004	18-Jul-19	18-Jul-19	26603	136804	0.19446	0.3022826	-0.000281	0.64425
210	REG-2019-0035 . POF . B . 132-144 hr . 1 . 200	004	18-Jul-19	18-Jul-19	23643	120237	0.19664	0.3022826	-0.000281	0.65145
211	REG-2019-0035 . POF . C . 132-144 hr . 1 . 201	004	18-Jul-19	18-Jul-19	19178	112845	0.16995	0.3022826	-0.000281	0.56316
212	REG-2019-0035 . POF . D . 132-144 hr . 1 . 202	004	18-Jul-19	18-Jul-19	19073	126213	0.15112	0.3022826	-0.000281	0.50085
213	REG-2019-0035 . POF . E . 132-144 hr . 1 . 203	004	18-Jul-19	19-Jul-19	21655	133186	0.16259	0.3022826	-0.000281	0.53881
214	REG-2019-0035 . POF . F . 132-144 hr . 1 . 204	004	18-Jul-19	19-Jul-19	17658	137411	0.12850	0.3022826	-0.000281	0.42604
215	REG-2019-0035 . POF . G . 132-144 hr . 1 . 205	004	18-Jul-19	19-Jul-19	14189	110146	0.12882	0.3022826	-0.000281	0.42708
216	REG-2019-0035 . POF . H . 132-144 hr . 1 . 206	004	18-Jul-19	19-Jul-19	23923	129084	0.18533	0.3022826	-0.000281	0.61404
217	REG-2019-0035 . POF . A . 144-156 hr . 1 . 213	004	18-Jul-19	19-Jul-19	15001	121482	0.12348	0.3022826	-0.000281	0.40942
218	REG-2019-0035 . POF . B . 144-156 hr . 1 . 214	004	18-Jul-19	19-Jul-19	13336	150257	0.08876	0.3022826	-0.000281	0.29455
219	REG-2019-0035 . POF . C . 144-156 hr . 1 . 215	004	18-Jul-19	19-Jul-19	15624	127462	0.12258	0.3022826	-0.000281	0.40643
220	REG-2019-0035 . POF . D . 144-156 hr . 1 . 216	004	18-Jul-19	19-Jul-19	13607	144086	0.09444	0.3022826	-0.000281	0.31335

### Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
221	REG-2019-0035 . POF . E . 144-156 hr . 1 . 217	004	18-Jul-19	19-Jul-19	13437	123083	0.10917	0.3022826	-0.000281	0.36207
222	REG-2019-0035 . POF . F . 144-156 hr . 1 . 218	004	18-Jul-19	19-Jul-19	13815	124673	0.11081	0.3022826	-0.000281	0.36751
223	REG-2019-0035 . POF . G . 144-156 hr . 1 . 219	004	18-Jul-19	19-Jul-19	12755	120546	0.10581	0.3022826	-0.000281	0.35097
224	REG-2019-0035 . POF . H . 144-156 hr . 1 . 220	004	18-Jul-19	19-Jul-19	25791	134934	0.19113	0.3022826	-0.000281	0.63323
248	REG-2019-0035 . POF . A . 156-168 hr . 1 . 227	005	19-Jul-19	20-Jul-19	76620	150812	0.50805	0.3069103	0.002515019	1.64718
249	REG-2019-0035 . POF . B . 156-168 hr . 1 . 228	005	19-Jul-19	20-Jul-19	58260	149487	0.38973	0.3069103	0.002515019	1.26166
250	REG-2019-0035 . POF . C . 156-168 hr . 1 . 229	005	19-Jul-19	20-Jul-19	56400	168878	0.33397	0.3069103	0.002515019	1.07996
251	REG-2019-0035 . POF . D . 156-168 hr . 1 . 230	005	19-Jul-19	20-Jul-19	42328	141594	0.29894	0.3069103	0.002515019	0.96584
252	REG-2019-0035 . POF . E . 156-168 hr . 1 . 231	005	19-Jul-19	20-Jul-19	42884	169217	0.25342	0.3069103	0.002515019	0.81753
253	REG-2019-0035 . POF . F . 156-168 hr . 1 . 232	005	19-Jul-19	20-Jul-19	37466	145889	0.25681	0.3069103	0.002515019	0.82857
254	REG-2019-0035 . POF . G . 156-168 hr . 1 . 233	005	19-Jul-19	20-Jul-19	34949	134318	0.26020	0.3069103	0.002515019	0.83961
255	REG-2019-0035 . POF . H . 156-168 hr . 1 . 234	005	19-Jul-19	20-Jul-19	38260	142650	0.26821	0.3069103	0.002515019	0.86570
262	REG-2019-0035 . SKPUF . 0 ng . 6 hr . 1	005	19-Jul-19	20-Jul-19	2236	140540	0.01591	0.3069103	0.002515019	0.04364
263	REG-2019-0035 . SKPUF . 0 ng . 6 hr . 2	005	19-Jul-19	20-Jul-19	5977	151107	0.03955	0.3069103	0.002515019	0.12068
264	REG-2019-0035 . SKPUF . 0 ng . 6 hr . 3	005	19-Jul-19	20-Jul-19	1607	130083	0.01236	0.3069103	0.002515019	0.03207
265	REG-2019-0035 . SKPUF . 3 ng . 6 hr . 1	005	19-Jul-19	20-Jul-19	118558	133946	0.88512	0.3069103	0.002515019	2.87576
266	REG-2019-0035 . SKPUF . 3 ng . 6 hr . 2	005	19-Jul-19	20-Jul-19	124849	140711	0.88727	0.3069103	0.002515019	2.88278
267	REG-2019-0035 . SKPUF . 3 ng . 6 hr . 3	005	19-Jul-19	20-Jul-19	121278	138989	0.87257	0.3069103	0.002515019	2.83488
268	REG-2019-0035 . SKPUF . 10 ng . 6 hr . 1	005	19-Jul-19	20-Jul-19	46560	16228	2.86915	0.3069103	0.002515019	9.34031
269	REG-2019-0035 . SKPUF . 10 ng . 6 hr . 2	005	19-Jul-19	20-Jul-19	37211	12330	3.01807	0.3069103	0.002515019	9.82553
270	REG-2019-0035 . SKPUF . 10 ng . 6 hr . 3	005	19-Jul-19	20-Jul-19	47805	16317	2.92982	0.3069103	0.002515019	9.53797
271	REG-2019-0035 . SKPUF . 30 ng . 6 hr . 1	005	19-Jul-19	20-Jul-19	137966	16012	8.61657	0.3069103	0.002515019	28.06700
272	REG-2019-0035 . SKPUF . 30 ng . 6 hr . 2	005	19-Jul-19	20-Jul-19	151211	15030	10.06055	0.3069103	0.002515019	32.77189
273	REG-2019-0035 . SKPUF . 30 ng . 6 hr . 3	005	19-Jul-19	20-Jul-19	143700	14474	9.92792	0.3069103	0.002515019	32.33977
274	REG-2019-0035 . SKPUF . 0 ng . 12 hr . 1	005	19-Jul-19	20-Jul-19	1203	134652	0.00894	0.3069103	0.002515019	0.02092



# Raw Data Table for PUF Sample Analysis

EEAL ID (89311-)	Sample ID	Set No.	Extraction Date	Analysis Date	Peak Area	IS Peak Area	Area Ratio	Curve Constants		Dicamba (ng/PUF)
								m	b	
275	REG-2019-0035 . SKPUF . 0 ng . 12 hr . 2	005	19-Jul-19	20-Jul-19	2500	134606	0.01857	0.3069103	0.002515019	0.05233
276	REG-2019-0035 . SKPUF . 0 ng . 12 hr . 3	005	19-Jul-19	20-Jul-19	916	148980	0.00615	0.3069103	0.002515019	0.01183
277	REG-2019-0035 . SKPUF . 3 ng . 12 hr . 1	005	19-Jul-19	20-Jul-19	112508	113592	0.99046	0.3069103	0.002515019	3.21899
278	REG-2019-0035 . SKPUF . 3 ng . 12 hr . 2	005	19-Jul-19	20-Jul-19	129819	142479	0.91114	0.3069103	0.002515019	2.96056
279	REG-2019-0035 . SKPUF . 3 ng . 12 hr . 3	005	19-Jul-19	20-Jul-19	114551	126218	0.90756	0.3069103	0.002515019	2.94890
280	REG-2019-0035 . SKPUF . 10 ng . 12 hr . 1	005	19-Jul-19	20-Jul-19	43017	15711	2.73799	0.3069103	0.002515019	8.91295
281	REG-2019-0035 . SKPUF . 10 ng . 12 hr . 2	005	19-Jul-19	20-Jul-19	37933	11915	3.18374	0.3069103	0.002515019	10.36531
282	REG-2019-0035 . SKPUF . 10 ng . 12 hr . 3	005	19-Jul-19	20-Jul-19	41640	14647	2.84292	0.3069103	0.002515019	9.25482
283	REG-2019-0035 . SKPUF . 30 ng . 12 hr . 1	005	19-Jul-19	20-Jul-19	129238	14186	9.11035	0.3069103	0.002515019	29.67588
284	REG-2019-0035 . SKPUF . 30 ng . 12 hr . 2	005	19-Jul-19	20-Jul-19	144480	15776	9.15817	0.3069103	0.002515019	29.83169
285	REG-2019-0035 . SKPUF . 30 ng . 12 hr . 3	005	19-Jul-19	20-Jul-19	144478	16172	8.93391	0.3069103	0.002515019	29.10100
256	REG-2019-0035 . TSPUF . 0 ng . 1	005	19-Jul-19	20-Jul-19	0	123395	0.00000	0.3069103	0.002515019	0.00000
257	REG-2019-0035 . TSPUF . 0 ng . 2	005	19-Jul-19	20-Jul-19	0	137374	0.00000	0.3069103	0.002515019	0.00000
258	REG-2019-0035 . TSPUF . 0 ng . 3	005	19-Jul-19	20-Jul-19	0	177886	0.00000	0.3069103	0.002515019	0.00000
259	REG-2019-0035 . TSPUF . 30 ng . 1	005	19-Jul-19	20-Jul-19	141562	15652	9.04432	0.3069103	0.002515019	29.46075
260	REG-2019-0035 . TSPUF . 30 ng . 2	005	19-Jul-19	20-Jul-19	120835	14239	8.48642	0.3069103	0.002515019	27.64294
261	REG-2019-0035 . TSPUF . 30 ng . 3	005	19-Jul-19	20-Jul-19	114606	11768	9.73842	0.3069103	0.002515019	31.72230

## **Appendix H. Significant Dates Table**

The *Significant Dates Table* contains the sampling, extraction, and analysis dates for each sample. A description of the columns is presented below.

### **A. Sample ID**

The field ID which identifies study, sample type, location or distance, timing, and replicate for each sample analyzed.

### **B. Sampling Date**

The date on which the samples were collected in the field. For field QC and transit stability samples, this date reflects the date the samples were fortified.

### **C. Extraction Date**

The date the samples were extracted in the analytical laboratory.

### **D. Analysis Date**

The date the analytical results were acquired from the instrument.

### **E. Days between Sampling and Extraction**

The number of days between sample collection in the field and extraction in the laboratory.

### **F. Days between Extraction and Analysis**

The number of days between extraction and the acquisition of the analytical results.

## Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . PCM . 015 . PRE . 1 . 001	30-Jun-19	15-Jul-19	16-Jul-19	15	1
REG-2019-0035 . PCM . 015 . PRE . 2 . 002	30-Jun-19	15-Jul-19	16-Jul-19	15	1
REG-2019-0035 . FPS . A . APP . 1 . 003	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . A . APP . 2 . 004	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . A . APP . 3 . 005	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . A . APP . 4 . 006	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . B . APP . 1 . 007	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . B . APP . 2 . 008	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . B . APP . 3 . 009	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . B . APP . 4 . 010	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . C . APP . 1 . 011	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . C . APP . 2 . 012	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . C . APP . 3 . 013	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . C . APP . 4 . 014	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . D . APP . 1 . 015	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . D . APP . 2 . 016	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . D . APP . 3 . 017	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . FPS . D . APP . 4 . 018	02-Jul-19	18-Jul-19	18-Jul-19	16	0
REG-2019-0035 . TNK . A . PRE-APP . 1 . 019	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . TNK . A . PRE-APP . 2 . 020	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . TNK . A . PRE-APP . 3 . 021	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . TNK . A . POST-APP . 1 . 022	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . TNK . A . POST-APP . 2 . 023	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . TNK . A . POST-APP . 3 . 024	02-Jul-19	19-Jul-19	19-Jul-19	17	0
REG-2019-0035 . PCM . 015 . 0-6 hr . 1 . 025	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 033 . 0-6 hr . 1 . 026	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 055 . 0-6 hr . 1 . 027	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 090 . 0-6 hr . 1 . 028	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 150 . 0-6 hr . 1 . 029	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 150 . 0-6 hr . 2 . 030	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . A . 0-6 hr . 1 . 031	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . B . 0-6 hr . 1 . 032	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . C . 0-6 hr . 1 . 033	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . D . 0-6 hr . 1 . 034	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . E . 0-6 hr . 1 . 035	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . F . 0-6 hr . 1 . 036	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . G . 0-6 hr . 1 . 037	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . H . 0-6 hr . 1 . 038	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 015 . 6-12 . 1 . 039	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 033 . 6-12 . 1 . 040	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 055 . 6-12 . 1 . 041	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 090 . 6-12 . 1 . 042	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 150 . 6-12 . 1 . 043	02-Jul-19	15-Jul-19	16-Jul-19	13	1

## Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . PCM . 150 . 6-12 . 2 . 044	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . A . 6-12 hr . 1 . 045	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . B . 6-12 hr . 1 . 046	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . C . 6-12 hr . 1 . 047	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . D . 6-12 hr . 1 . 048	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . E . 6-12 hr . 1 . 049	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . F . 6-12 hr . 1 . 050	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . G . 6-12 hr . 1 . 051	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . POF . H . 6-12 hr . 1 . 052	02-Jul-19	15-Jul-19	16-Jul-19	13	1
REG-2019-0035 . PCM . 015 . 12-24 hr . 1 . 053	03-Jul-19	15-Jul-19	16-Jul-19	12	1
REG-2019-0035 . PCM . 033 . 12-24 hr . 1 . 054	03-Jul-19	15-Jul-19	16-Jul-19	12	1
REG-2019-0035 . PCM . 055 . 12-24 hr . 1 . 055	03-Jul-19	15-Jul-19	16-Jul-19	12	1
REG-2019-0035 . PCM . 090 . 12-24 hr . 1 . 056	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . PCM . 150 . 12-24 hr . 1 . 057	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . PCM . 150 . 12-24 hr . 2 . 058	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . A . 12-24 hr . 1 . 059	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . B . 12-24 hr . 1 . 060	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . C . 12-24 hr . 1 . 061	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . D . 12-24 hr . 1 . 062	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . E . 12-24 hr . 1 . 063	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . F . 12-24 hr . 1 . 064	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . G . 12-24 hr . 1 . 065	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . H . 12-24 hr . 1 . 066	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . PCM . 015 . 24-36 hr . 1 . 067	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 033 . 24-36 hr . 1 . 068	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 055 . 24-36 hr . 1 . 069	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 090 . 24-36 hr . 1 . 070	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 150 . 24-36 hr . 1 . 071	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 150 . 24-36 hr . 2 . 072	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . POF . A . 24-36 hr . 1 . 073	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . B . 24-36 hr . 1 . 074	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . C . 24-36 hr . 1 . 075	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . D . 24-36 hr . 1 . 076	03-Jul-19	15-Jul-19	17-Jul-19	12	2
REG-2019-0035 . POF . E . 24-36 hr . 1 . 077	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . POF . F . 24-36 hr . 1 . 078	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . POF . G . 24-36 hr . 1 . 079	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . POF . H . 24-36 hr . 1 . 080	03-Jul-19	16-Jul-19	17-Jul-19	13	1
REG-2019-0035 . PCM . 015 . 36-48 hr . 1 . 081	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 033 . 36-48 hr . 1 . 082	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 055 . 36-48 hr . 1 . 083	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 090 . 36-48 hr . 1 . 084	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 36-48 hr . 1 . 085	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 36-48 hr . 2 . 086	04-Jul-19	16-Jul-19	17-Jul-19	12	1

## Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . POF . A . 36-48 hr . 1 . 087	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . B . 36-48 hr . 1 . 088	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . C . 36-48 hr . 1 . 089	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . D . 36-48 hr . 1 . 090	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . E . 36-48 hr . 1 . 091	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . F . 36-48 hr . 1 . 092	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . G . 36-48 hr . 1 . 093	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . H . 36-48 hr . 1 . 094	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 015 . 48-60 hr . 1 . 095	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 033 . 48-60 hr . 1 . 096	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 055 . 48-60 hr . 1 . 097	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 090 . 48-60 hr . 1 . 098	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 48-60 hr . 1 . 099	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 48-60 hr . 2 . 100	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . A . 48-60 hr . 1 . 101	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . B . 48-60 hr . 1 . 102	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . C . 48-60 hr . 1 . 103	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . D . 48-60 hr . 1 . 104	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . E . 48-60 hr . 1 . 105	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . F . 48-60 hr . 1 . 106	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . G . 48-60 hr . 1 . 107	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . POF . H . 48-60 hr . 1 . 108	04-Jul-19	16-Jul-19	17-Jul-19	12	1
REG-2019-0035 . PCM . 015 . 60-72 hr . 1 . 109	05-Jul-19	16-Jul-19	17-Jul-19	11	1
REG-2019-0035 . PCM . 033 . 60-72 hr . 1 . 110	05-Jul-19	16-Jul-19	17-Jul-19	11	1
REG-2019-0035 . PCM . 055 . 60-72 hr . 1 . 111	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . PCM . 090 . 60-72 hr . 1 . 112	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . PCM . 150 . 60-72 hr . 1 . 113	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . PCM . 150 . 60-72 hr . 2 . 114	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . POF . A . 60-72 hr . 1 . 115	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . POF . B . 60-72 hr . 1 . 116	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . POF . C . 60-72 hr . 1 . 117	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . POF . D . 60-72 hr . 1 . 118	05-Jul-19	16-Jul-19	18-Jul-19	11	2
REG-2019-0035 . POF . E . 60-72 hr . 1 . 119	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . F . 60-72 hr . 1 . 120	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . G . 60-72 hr . 1 . 121	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . H . 60-72 hr . 1 . 122	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 015 . 72-84 hr . 1 . 123	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 033 . 72-84 hr . 1 . 124	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 055 . 72-84 hr . 1 . 125	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 090 . 72-84 hr . 1 . 126	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 72-84 hr . 1 . 127	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 150 . 72-84 hr . 2 . 128	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . A . 72-84 hr . 1 . 129	05-Jul-19	17-Jul-19	18-Jul-19	12	1

## Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . POF . B . 72-84 hr . 1 . 130	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . C . 72-84 hr . 1 . 131	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . D . 72-84 hr . 1 . 132	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . E . 72-84 hr . 1 . 133	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . F . 72-84 hr . 1 . 134	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . G . 72-84 hr . 1 . 135	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . POF . H . 72-84 hr . 1 . 136	05-Jul-19	17-Jul-19	18-Jul-19	12	1
REG-2019-0035 . PCM . 015 . 84-96 hr . 1 . 137	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 033 . 84-96 hr . 1 . 138	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 055 . 84-96 hr . 1 . 139	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 090 . 84-96 hr . 1 . 140	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 84-96 hr . 1 . 141	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 84-96 hr . 2 . 142	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . A . 84-96 hr . 1 . 143	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . B . 84-96 hr . 1 . 144	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . C . 84-96 hr . 1 . 145	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . D . 84-96 hr . 1 . 146	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . E . 84-96 hr . 1 . 147	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . F . 84-96 hr . 1 . 148	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . G . 84-96 hr . 1 . 149	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . H . 84-96 hr . 1 . 150	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 015 . 96-108 hr . 1 . 151	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 033 . 96-108 hr . 1 . 152	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 055 . 96-108 hr . 1 . 153	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 090 . 96-108 hr . 1 . 154	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 96-108 hr . 1 . 155	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 96-108 hr . 2 . 156	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . A . 96-108 hr . 1 . 157	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . B . 96-108 hr . 1 . 158	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . C . 96-108 hr . 1 . 159	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . D . 96-108 hr . 1 . 160	06-Jul-19	17-Jul-19	18-Jul-19	11	1
REG-2019-0035 . POF . E . 96-108 hr . 1 . 161	06-Jul-19	18-Jul-19	18-Jul-19	12	0
REG-2019-0035 . POF . F . 96-108 hr . 1 . 162	06-Jul-19	18-Jul-19	18-Jul-19	12	0
REG-2019-0035 . POF . G . 96-108 hr . 1 . 163	06-Jul-19	18-Jul-19	18-Jul-19	12	0
REG-2019-0035 . POF . H . 96-108 hr . 1 . 164	06-Jul-19	18-Jul-19	18-Jul-19	12	0
REG-2019-0035 . PCM . 015 . 108-120 hr . 1 . 165	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . PCM . 033 . 108-120 hr . 1 . 166	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . PCM . 055 . 108-120 hr . 1 . 167	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . PCM . 090 . 108-120 hr . 1 . 168	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . PCM . 150 . 108-120 hr . 1 . 169	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . PCM . 150 . 108-120 hr . 2 . 170	07-Jul-19	17-Jul-19	18-Jul-19	10	1
REG-2019-0035 . POF . A . 108-120 hr . 1 . 171	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . B . 108-120 hr . 1 . 172	07-Jul-19	18-Jul-19	18-Jul-19	11	0

## Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . POF . C . 108-120 hr . 1 . 173	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . D . 108-120 hr . 1 . 174	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . E . 108-120 hr . 1 . 175	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . F . 108-120 hr . 1 . 176	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . G . 108-120 hr . 1 . 177	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . H . 108-120 hr . 1 . 178	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 015 . 120-132 hr . 1 . 179	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 033 . 120-132 hr . 1 . 180	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 055 . 120-132 hr . 1 . 181	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 090 . 120-132 hr . 1 . 182	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 150 . 120-132 hr . 1 . 183	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 150 . 120-132 hr . 2 . 184	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . A . 120-132 hr . 1 . 185	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . B . 120-132 hr . 1 . 186	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . C . 120-132 hr . 1 . 187	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . D . 120-132 hr . 1 . 188	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . E . 120-132 hr . 1 . 189	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . F . 120-132 hr . 1 . 190	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . G . 120-132 hr . 1 . 191	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . POF . H . 120-132 hr . 1 . 192	07-Jul-19	18-Jul-19	18-Jul-19	11	0
REG-2019-0035 . PCM . 015 . 132-144 hr . 1 . 193	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . PCM . 033 . 132-144 hr . 1 . 194	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . PCM . 055 . 132-144 hr . 1 . 195	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . PCM . 090 . 132-144 hr . 1 . 196	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . PCM . 150 . 132-144 hr . 1 . 197	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . PCM . 150 . 132-144 hr . 2 . 198	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . POF . A . 132-144 hr . 1 . 199	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . POF . B . 132-144 hr . 1 . 200	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . POF . C . 132-144 hr . 1 . 201	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . POF . D . 132-144 hr . 1 . 202	08-Jul-19	18-Jul-19	18-Jul-19	10	0
REG-2019-0035 . POF . E . 132-144 hr . 1 . 203	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . F . 132-144 hr . 1 . 204	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . G . 132-144 hr . 1 . 205	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . H . 132-144 hr . 1 . 206	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . PCM . 015 . 144-156 hr . 1 . 207	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . PCM . 033 . 144-156 hr . 1 . 208	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . PCM . 055 . 144-156 hr . 1 . 209	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . PCM . 090 . 144-156 hr . 1 . 210	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 144-156 hr . 1 . 211	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . PCM . 150 . 144-156 hr . 2 . 212	08-Jul-19	19-Jul-19	20-Jul-19	11	1
REG-2019-0035 . POF . A . 144-156 hr . 1 . 213	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . B . 144-156 hr . 1 . 214	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . C . 144-156 hr . 1 . 215	08-Jul-19	18-Jul-19	19-Jul-19	10	1

### Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . POF . D . 144-156 hr . 1 . 216	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . E . 144-156 hr . 1 . 217	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . F . 144-156 hr . 1 . 218	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . G . 144-156 hr . 1 . 219	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . POF . H . 144-156 hr . 1 . 220	08-Jul-19	18-Jul-19	19-Jul-19	10	1
REG-2019-0035 . PCM . 015 . 156-168 hr . 1 . 221	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . PCM . 033 . 156-168 hr . 1 . 222	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . PCM . 055 . 156-168 hr . 1 . 223	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . PCM . 090 . 156-168 hr . 1 . 224	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . PCM . 150 . 156-168 hr . 1 . 225	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . PCM . 150 . 156-168 hr . 2 . 226	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . A . 156-168 hr . 1 . 227	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . B . 156-168 hr . 1 . 228	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . C . 156-168 hr . 1 . 229	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . D . 156-168 hr . 1 . 230	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . E . 156-168 hr . 1 . 231	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . F . 156-168 hr . 1 . 232	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . G . 156-168 hr . 1 . 233	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . POF . H . 156-168 hr . 1 . 234	09-Jul-19	19-Jul-19	20-Jul-19	10	1
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 0 ng . 6 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 6 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 6 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 6 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 0 ng . 12 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 3 ng . 12 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 10 ng . 12 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . SKPUF . 30 ng . 12 hr . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1



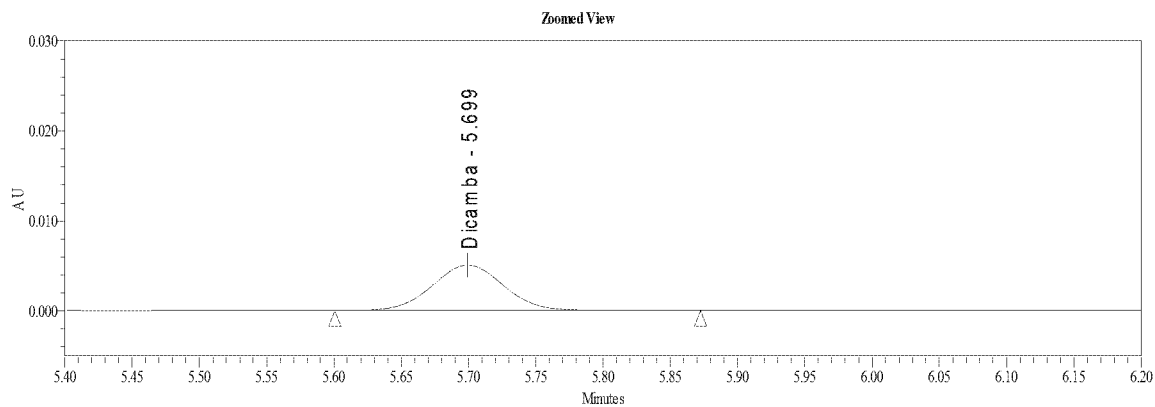
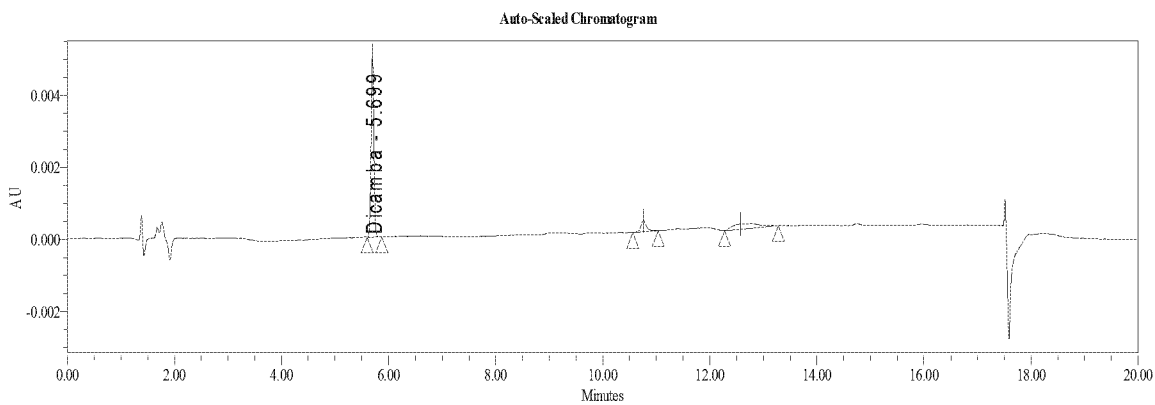
### Significant Dates Table

Sample ID	Sampling Date	Extraction Date	Analysis Date	Days between Sampling and Extraction	Days between Extraction and Analysis
REG-2019-0035 . TSPUF . 0 ng . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . TSPUF . 0 ng . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . TSPUF . 0 ng . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . TSPUF . 30 ng . 1	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . TSPUF . 30 ng . 2	02-Jul-19	19-Jul-19	20-Jul-19	17	1
REG-2019-0035 . TSPUF . 30 ng . 3	02-Jul-19	19-Jul-19	20-Jul-19	17	1

## Appendix I. Representative Chromatograms and Calibration Curve – Dicamba on Application Monitoring Filter Paper and Tank Mix

### Representative Chromatogram of 5.26 µg/g Calibration Standard

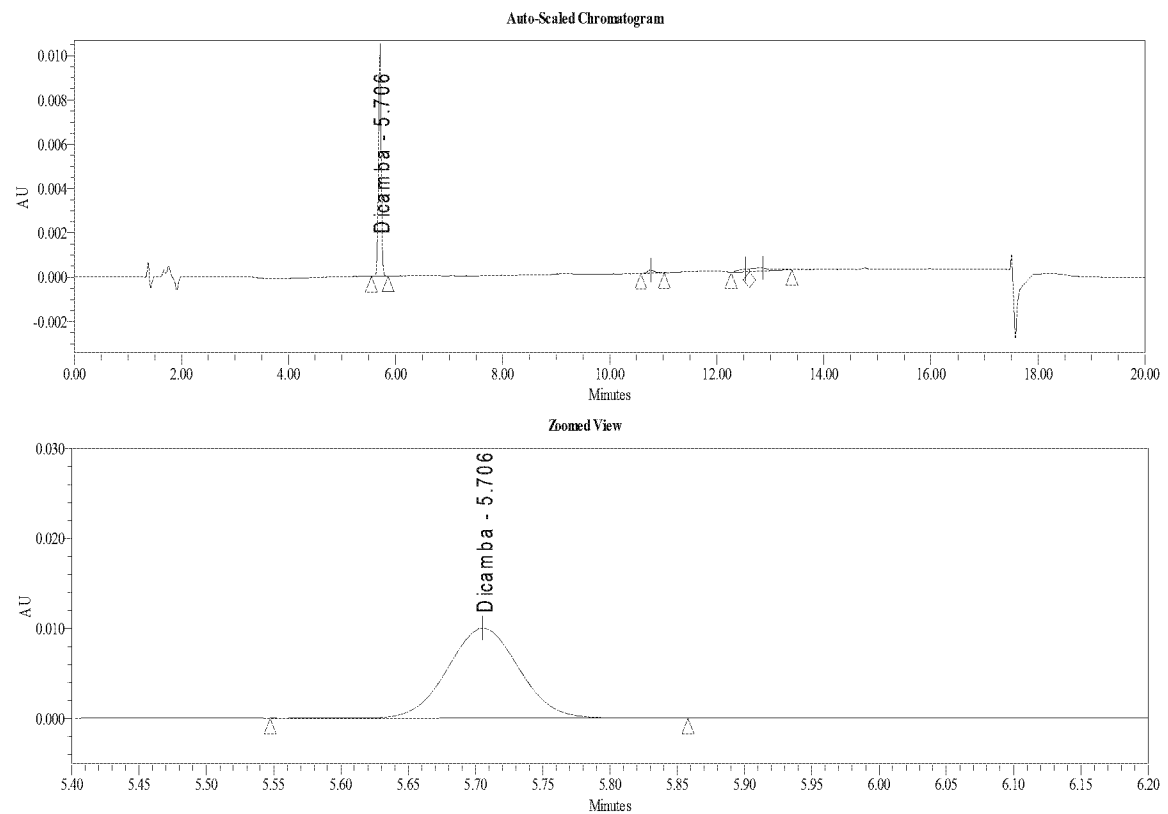
EEAL ID: CDG 17995 A-6



	SampleName	Result Id	Injection Id
1	CDG 17995 A-6	1225	1029

## Representative Chromatogram of 10.5 µg/g Calibration Standard

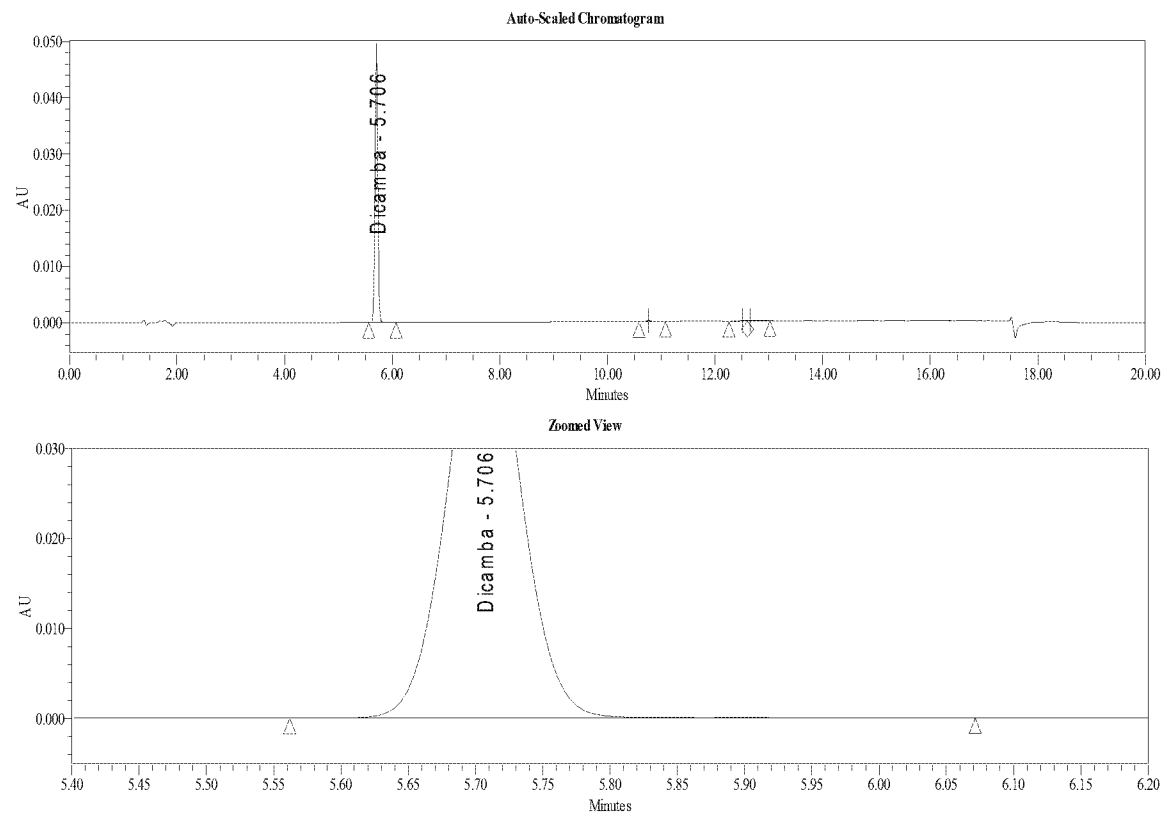
EEAL ID: CDG 17995 A-5



	SampleName	Result Id	Injection Id
1	CDG 17995 A-5	1230	1116

**Representative Chromatogram of 50.6 µg/g Calibration Standard**

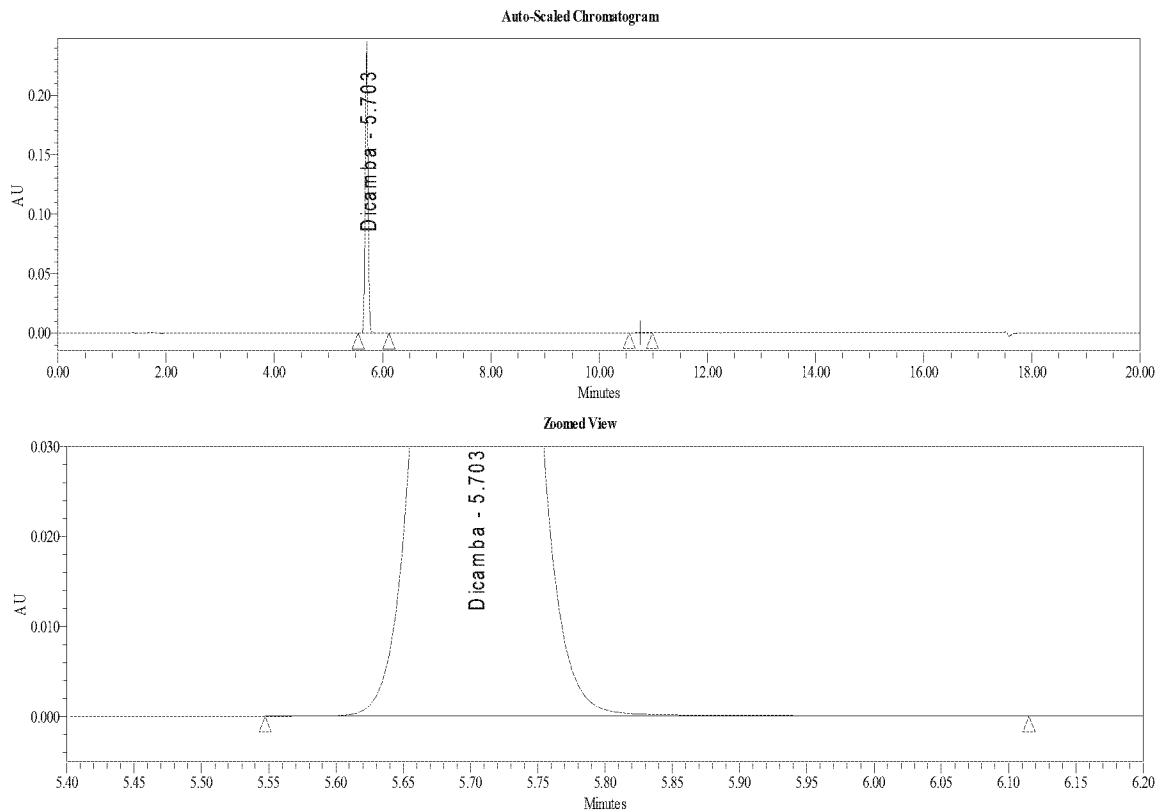
**EEAL ID: CDG 17995 A-3**



	SampleName	Result Id	Injection Id
1	CDG 17995 A-3	1227	1065

**Representative Chromatogram of 253 µg/g Calibration Standard**

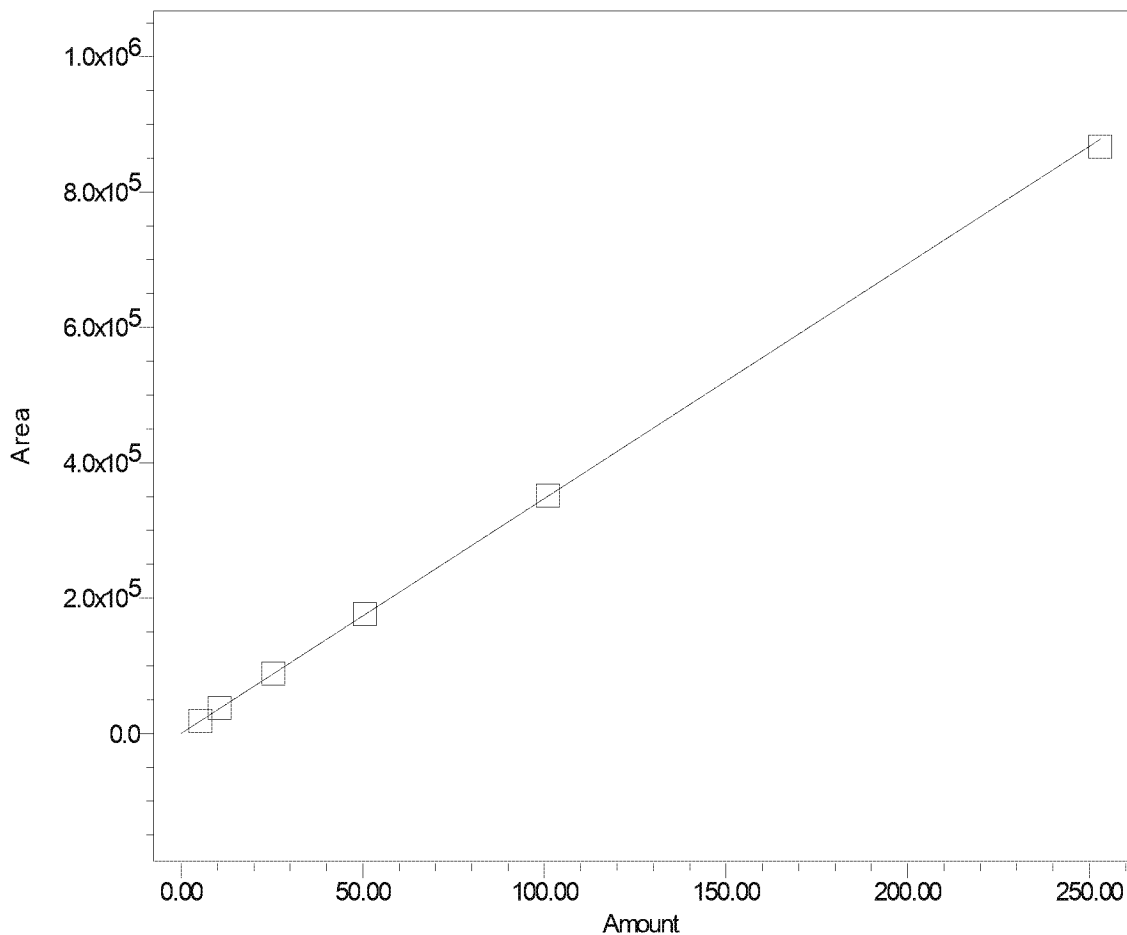
**EEAL ID: CDG 17995 A-1**



	SampleName	Result Id	Injection Id
1	CDG 17995 A-1	1226	1032

**Representative Calibration Curve**

## Calibration Plot



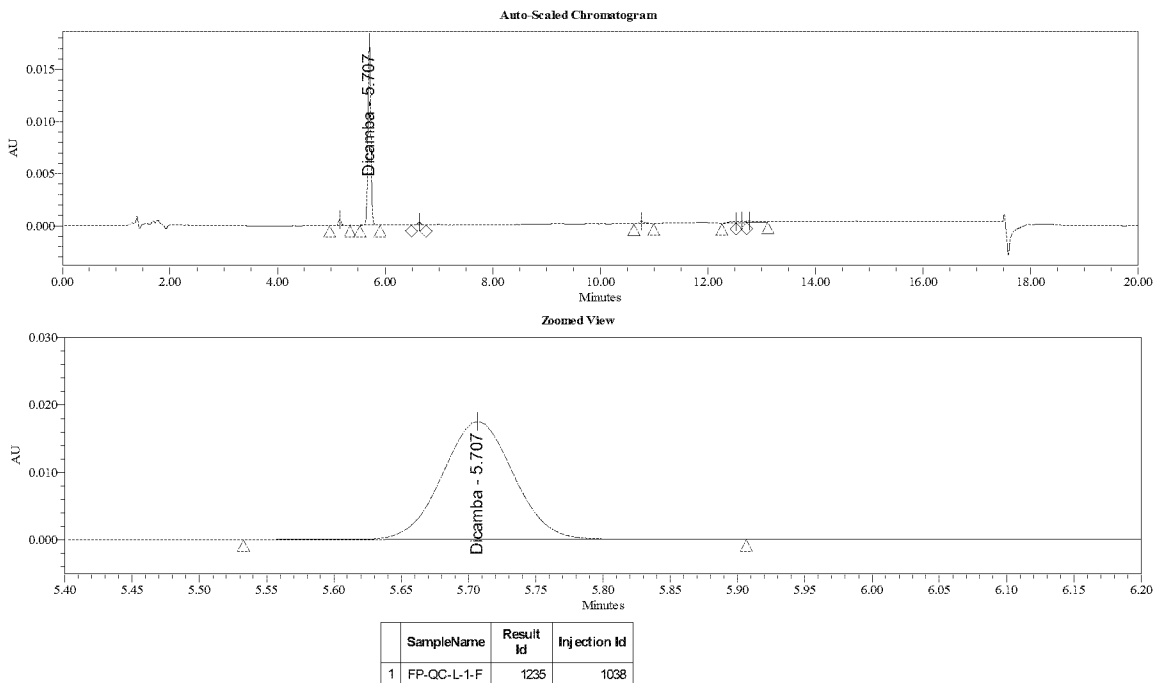
Name: Dicamba; Fit Type: Linear (1st Order); Cal Curve Id: 1224;  
Equation  $Y = 3.47e+003 X + 3.56e+002$ ; R 0.999956; R<sup>2</sup> 0.999913

### Peak: Dicamba

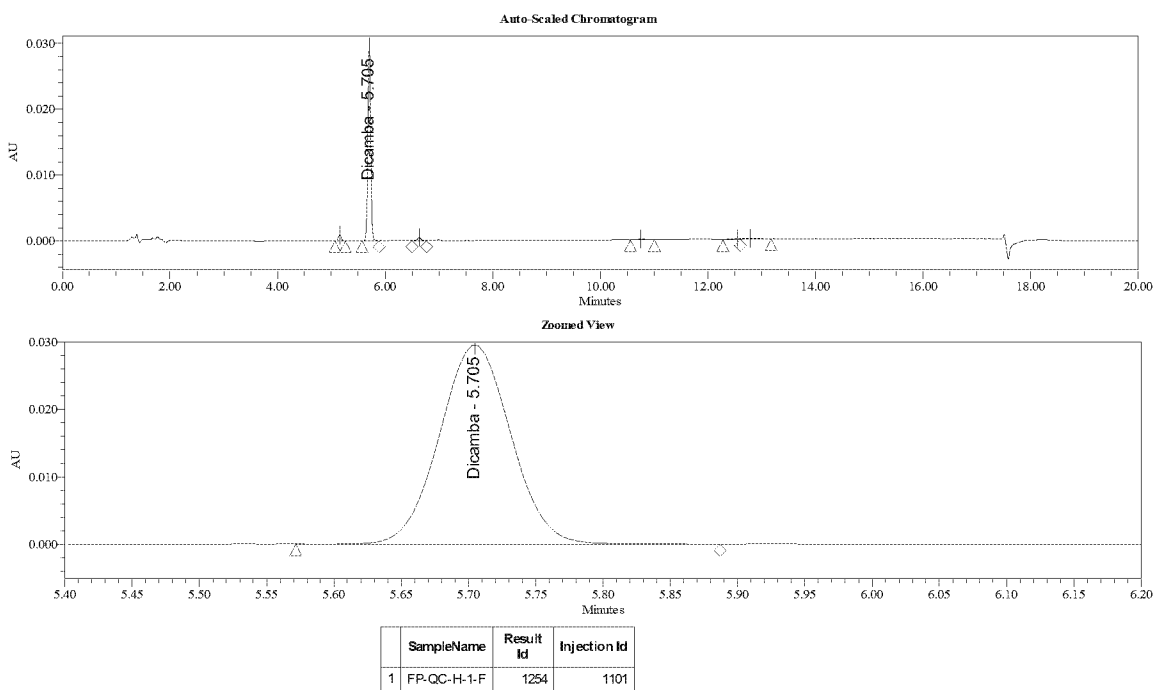
	Name	Level	X Value	Response	Calc. Value	% Deviation	Manual	Ignore
1	Dicamba		5.260000	18509.524807	5.231591	0.543	No	No
2	Dicamba		10.500000	37110.667414	10.592235	-0.871	No	No
3	Dicamba		25.300000	88509.745526	25.404881	-0.413	No	No
4	Dicamba		50.600000	176431.977087	50.743097	-0.282	No	No
5	Dicamba		101.000000	351491.642681	101.193359	-0.191	No	No
6	Dicamba		253.000000	867479.483010	249.895356	1.242	No	No

## Appendix J. Representative Chromatograms – Dicamba on Application Monitoring Filter Paper

**EEAL ID: FP-QC-L-1-F; Target Fortification: 0.516 mg/filter;  
Recovery: 103%**



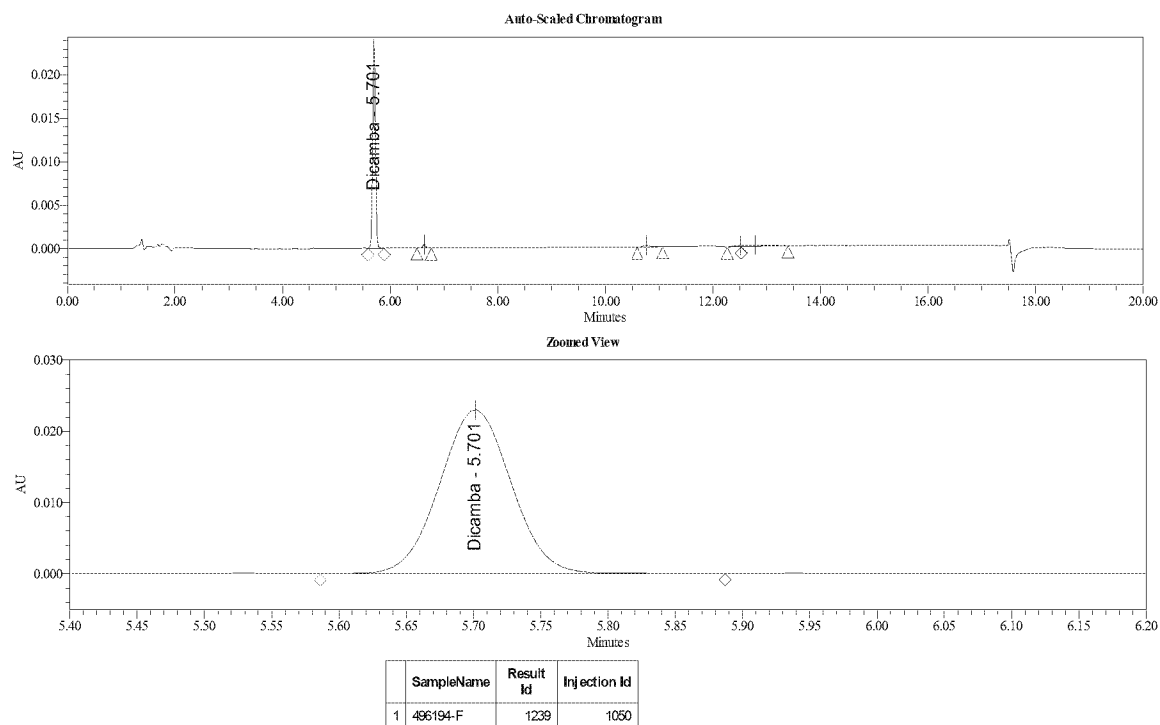
**EEAL ID: FP-QC-H-1-F; Target Fortification: 0.860 mg/filter;  
Recovery: 103%**



# Representative Chromatograms of Treated Application Monitoring Filter Paper

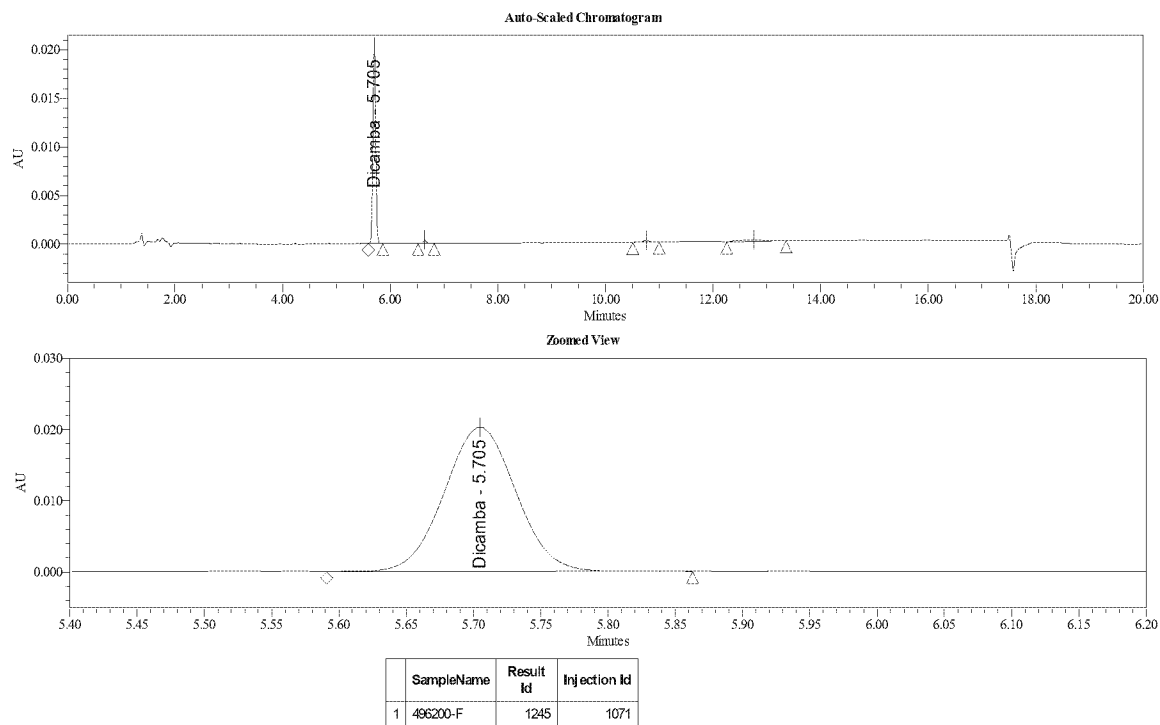
EEAL ID: 496194-F

Sample ID: REG-2019-0035 . FPS . A . APP . 2 . 004



EEAL ID: 496200-F

Sample ID: REG-2019-0035 . FPS . B . APP . 4 . 010

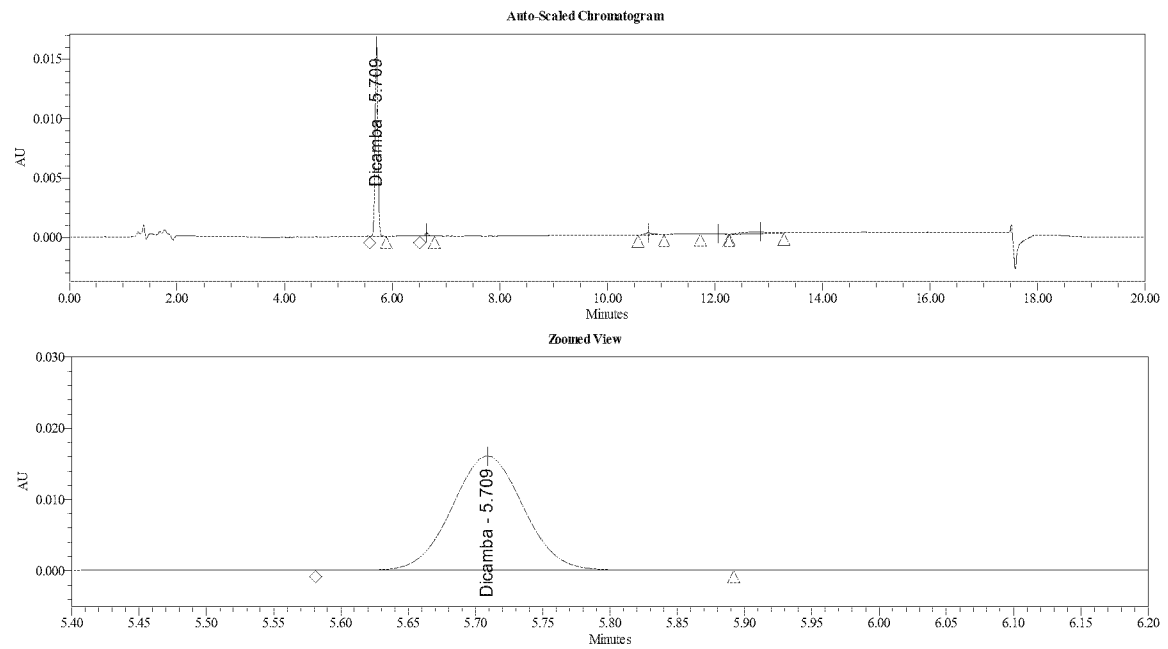




**Representative Chromatograms of Treated Application Monitoring Filter Paper (cont'd)**

**EEAL ID: 496203-F**

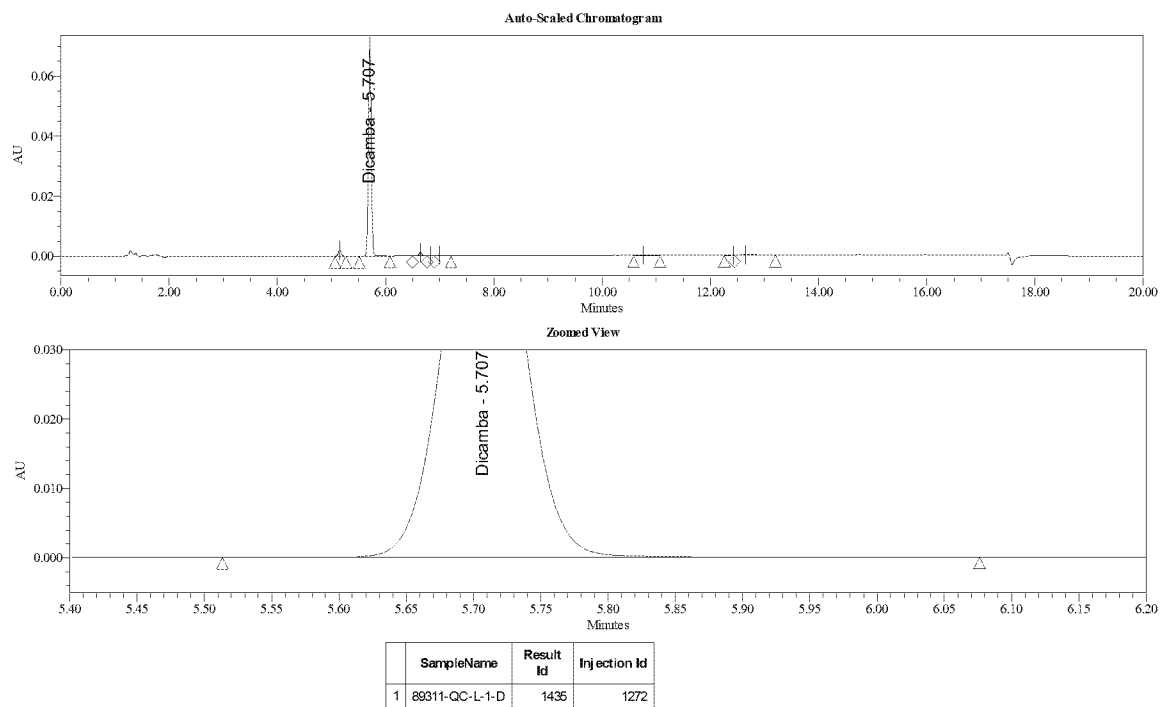
**Sample ID: REG-2019-0035 . FPS . C . APP . 3 . 013**



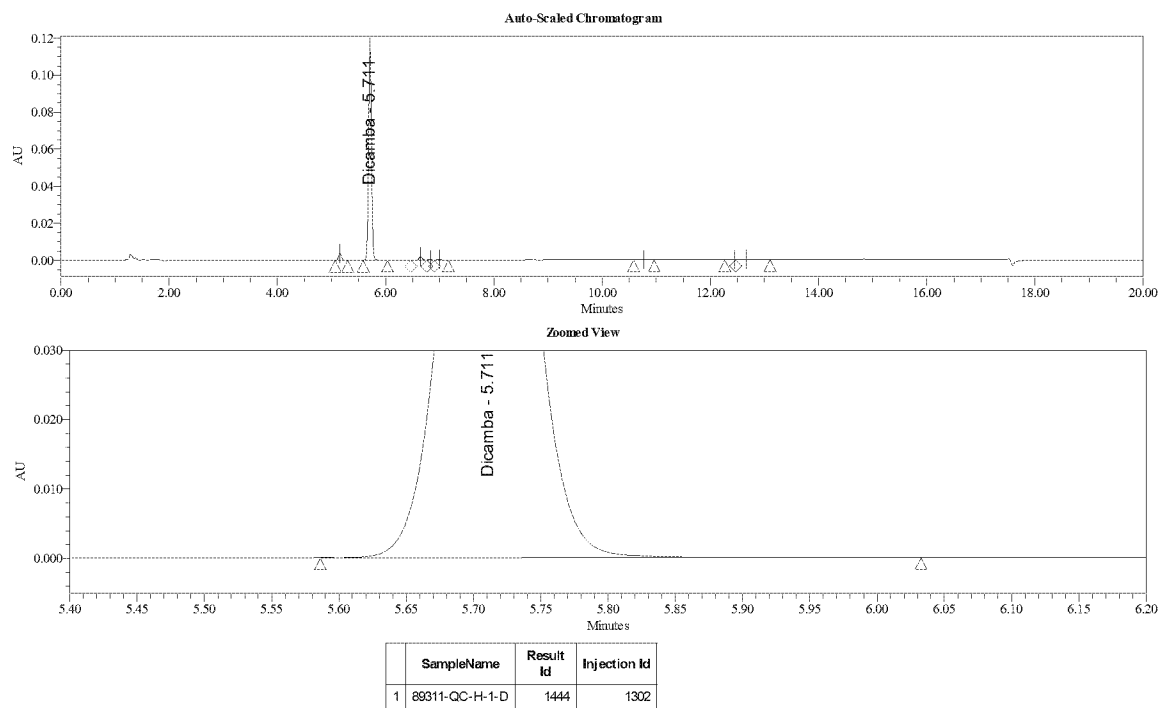
	SampleName	Result Id	Injection Id
1	496203-F	1248	1080

## Appendix K. Representative Chromatograms – Dicamba in Tank Mix

**EEAL ID: 89311-QC-L-1-D; Target Fortification: 0.30 wt% dicamba;  
Recovery: 99%**



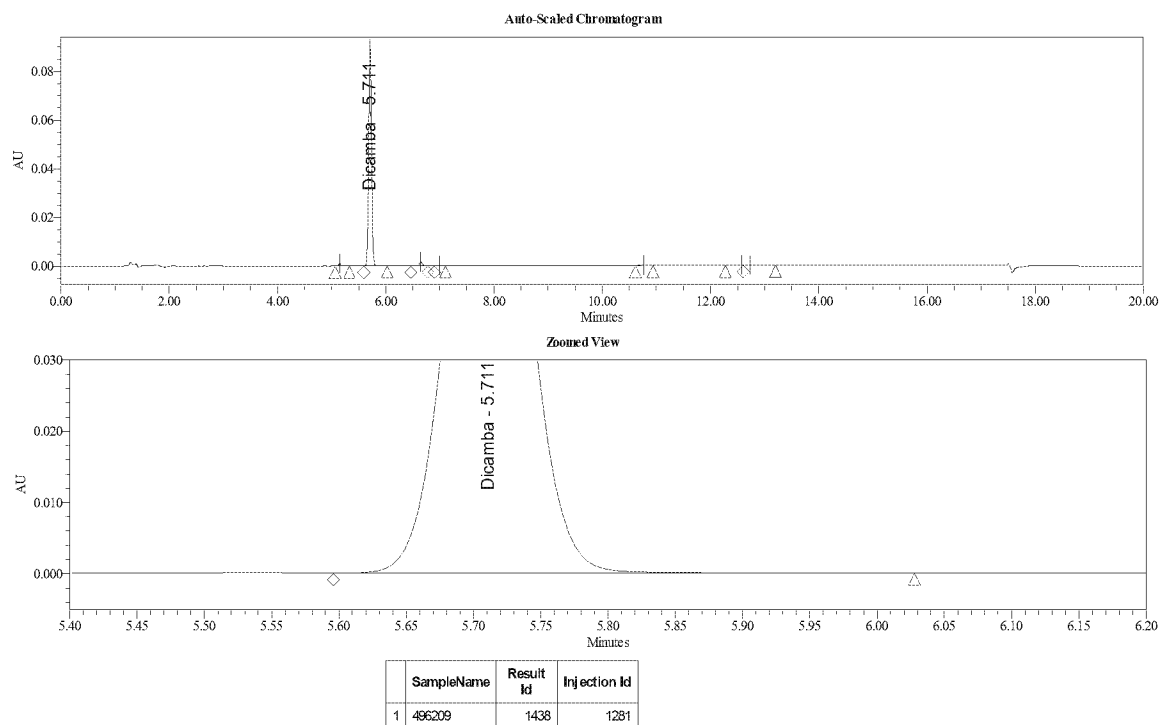
**EEAL ID: 89311-QC-H-1-D; Target Fortification: 0.50 wt% dicamba;  
Recovery: 99%**



# Representative Chromatograms of Tank Mix

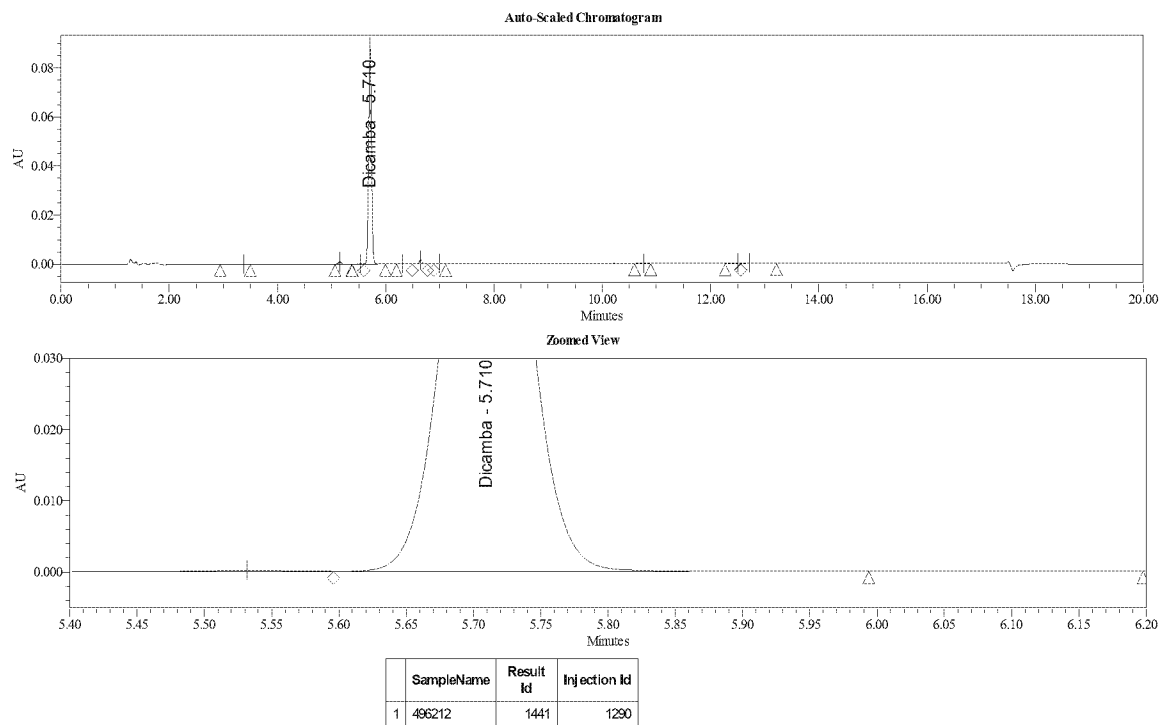
EEAL ID: 496209

Sample ID: REG-2019-0035 . TNK . A . PRE-APP . 1 . 019



EEAL ID: 496212

Sample ID: REG-2019-0035 . TNK . A . POST-APP . 1 . 022



## **Appendix L. Representative Chromatograms and Calibration Curve – Dicamba on PUF**

The following section contains representative chromatograms generated from the LC-MS/MS data system. Tables on the pages preceding the chromatograms for each analyte present a key to the identification of each of the sample chromatograms; it contains the sample number (based on the analysis set), the identification of the sample, and a brief description of the sample. The sample chromatograms are intended to provide the reader with examples of the analyte peaks and the background in the area of the analysis. This is readily seen in the sample chromatograms. If the reader wishes to calculate the concentration of the analyte in a given sample, use of the raw data tables is necessary. In the raw data table in Appendix G, the curve constants and all the necessary information for calculation of concentrations according to the equations provided in Appendix G are available. Each of the sample chromatograms contains the analysis of the sample (control, fortified or treated) on the top of the page, with the analysis of the corresponding internal standard on the bottom of the page. A  $^{13}\text{C}$ -labeled internal standard for each of the analytes was used in the analysis.

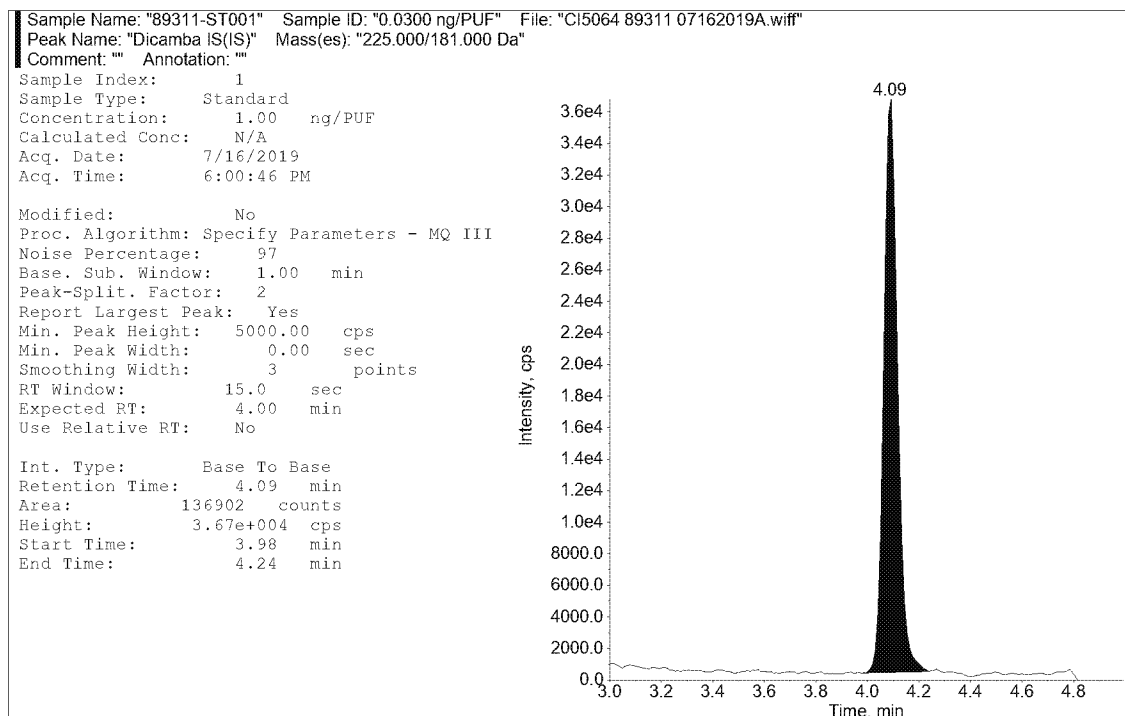
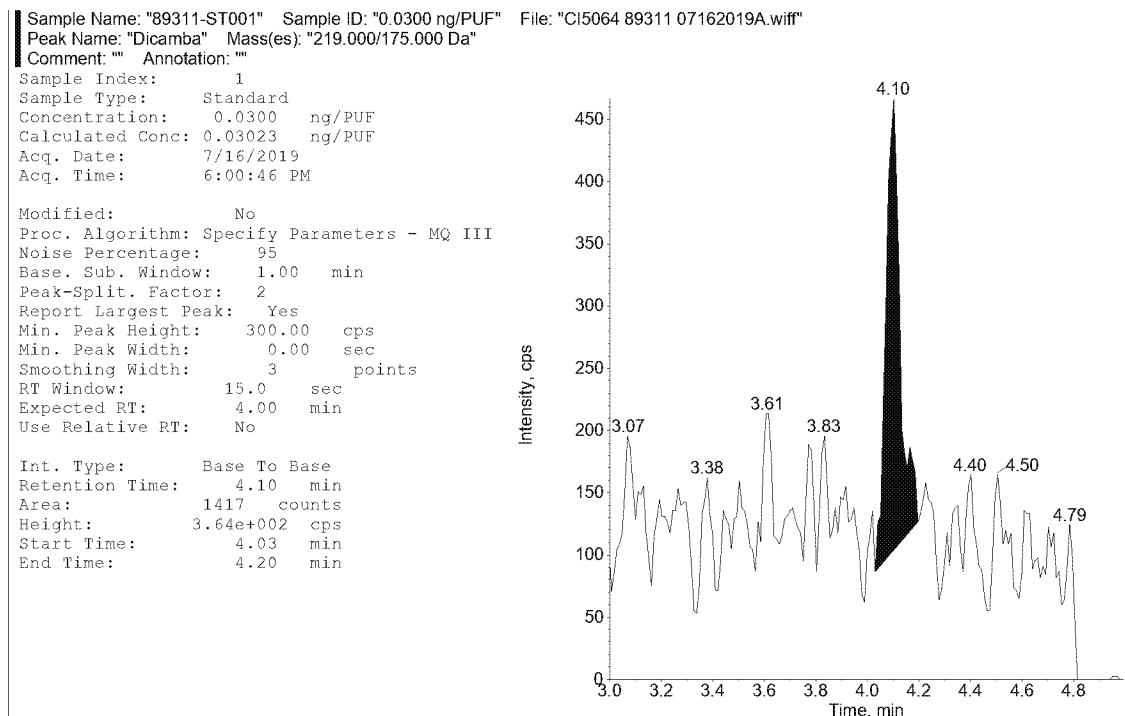
## L.1 Representative Chromatograms and Calibration Curve – Dicamba on PUF

### Representative Chromatograms of Standards and Calibration Curve

EEAL ID (89311-)	Sample ID	Sample Comment	Set ID
ST001	0.0300 ng/PUF Standard	Standard @ 0.0300 ng/PUF	001
ST005	0.300 ng/PUF Standard	Standard @ 0.300 ng/PUF	
ST009	3.00 ng/PUF Standard	Standard @ 3.00 ng/PUF	
ST010	7.50 ng/PUF Standard	Standard @ 7.50 ng/PUF	
Calibration Curve			

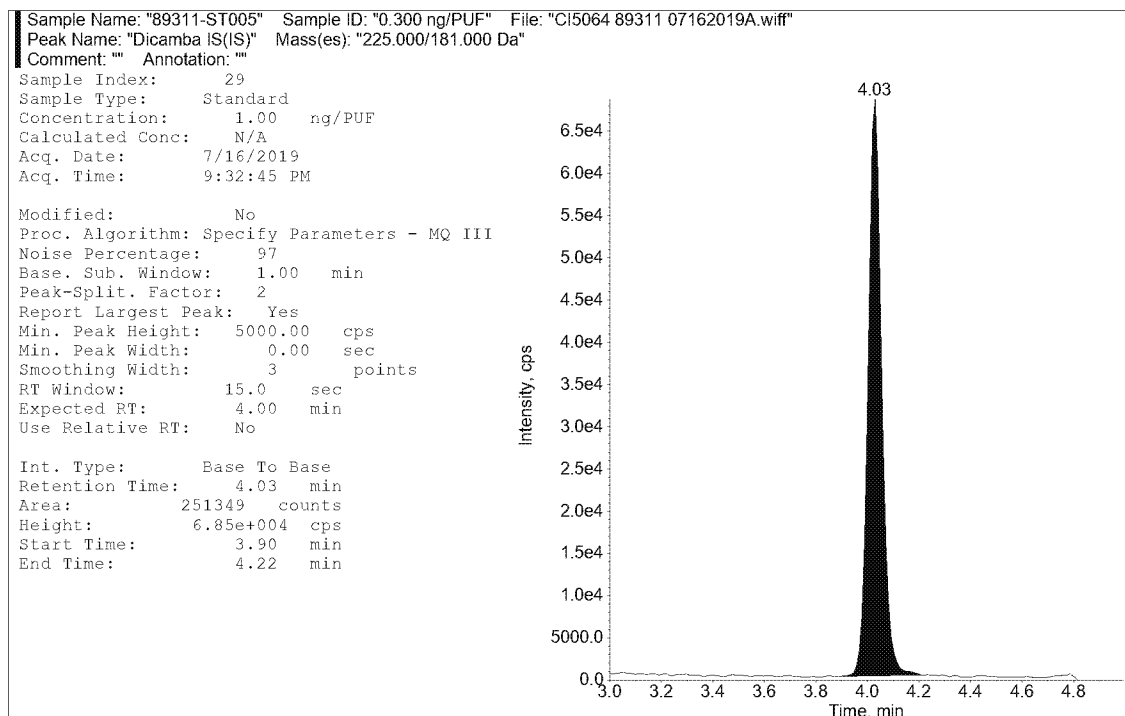
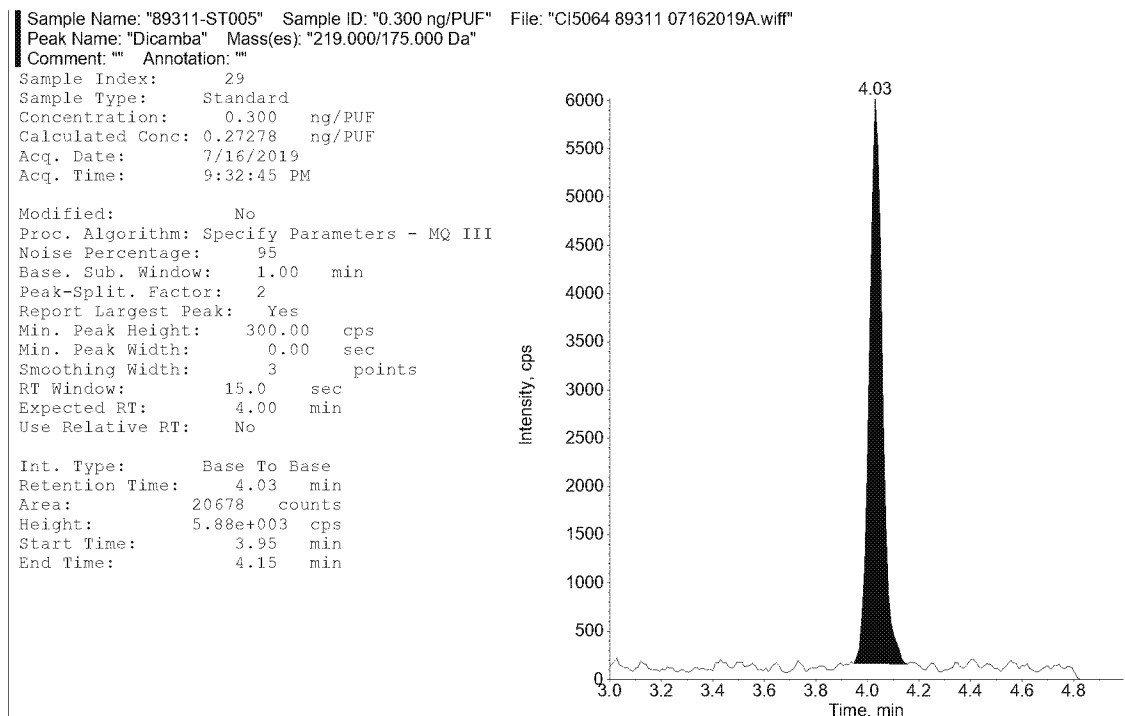
**Representative Chromatogram of 0.0300 ng/PUF Standard – Dicamba on PUF**

**EEAL ID: 89311-ST001**



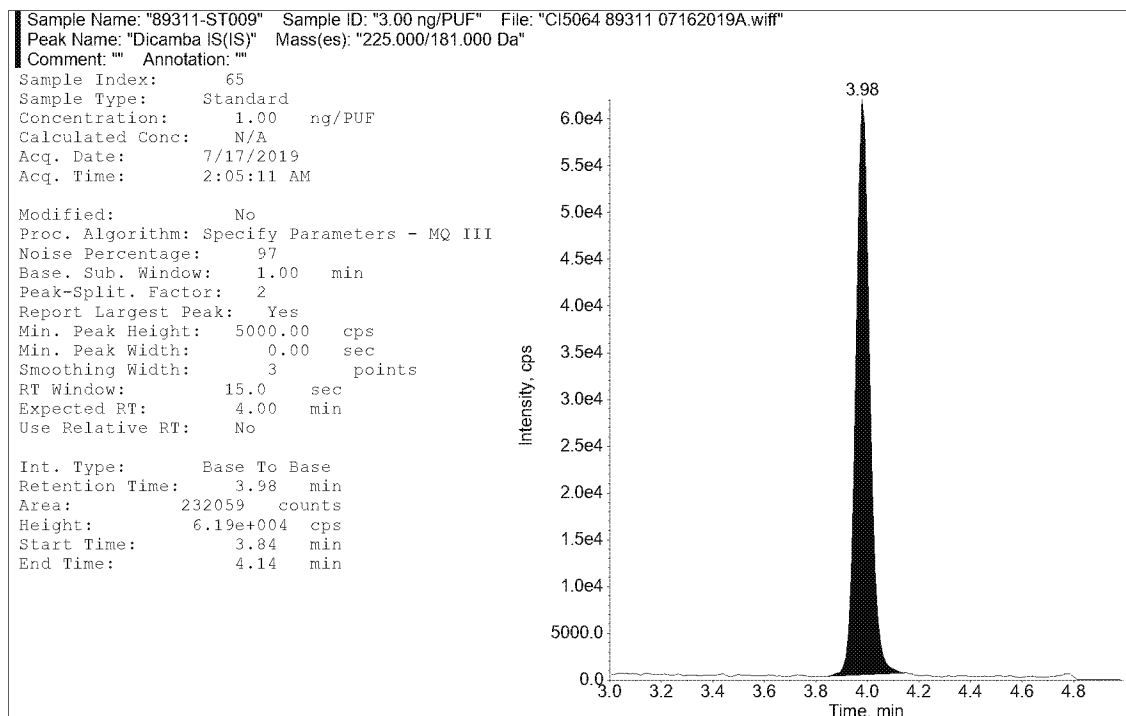
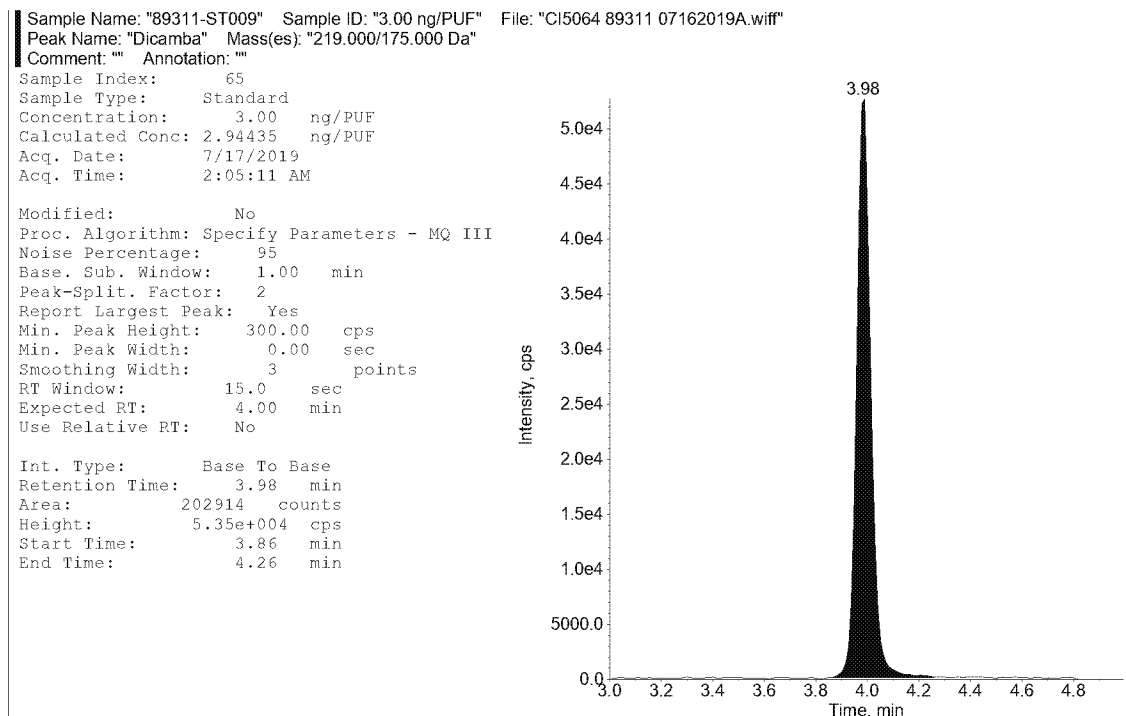
**Representative Chromatogram of 0.300 ng/PUF Standard – Dicamba on PUF**

**EEAL ID: 89311-ST005**



**Representative Chromatogram of 3.00 ng/PUF Standard – Dicamba on PUF**

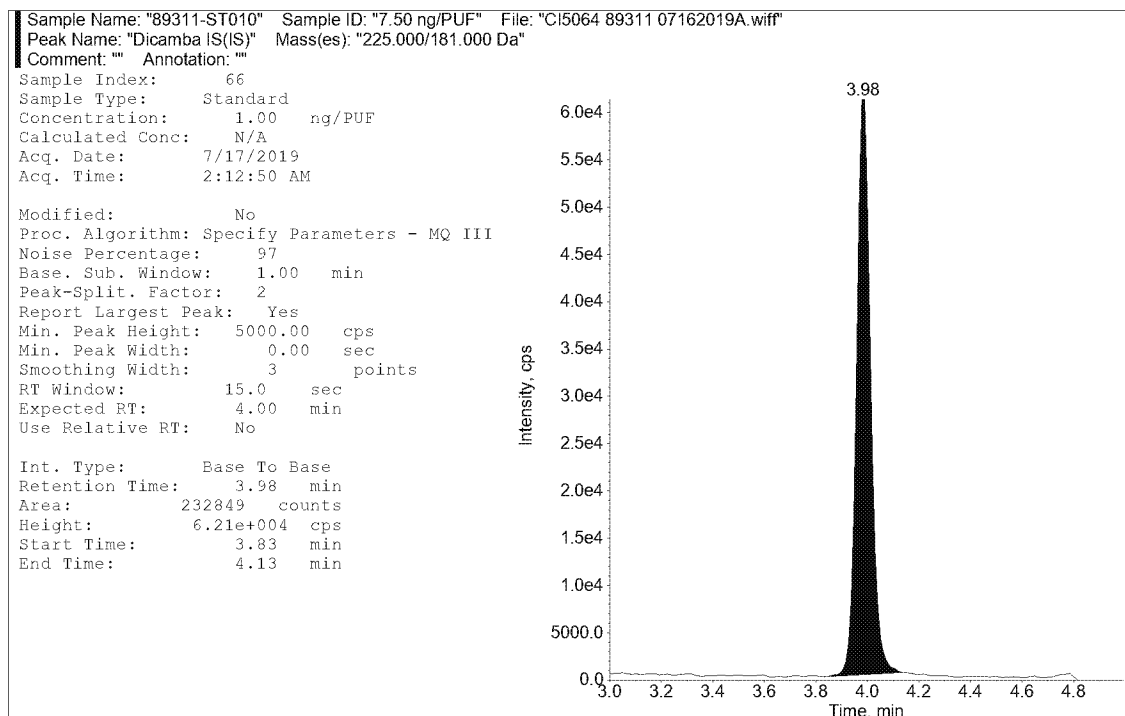
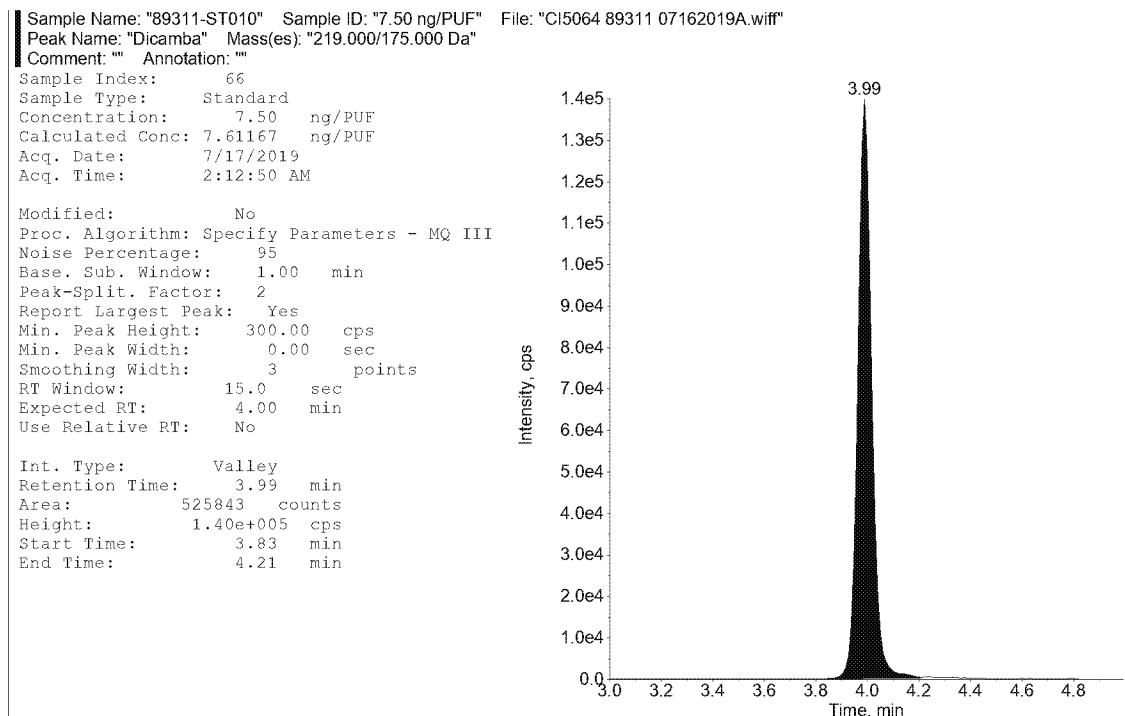
**EEAL ID: 89311-ST009**



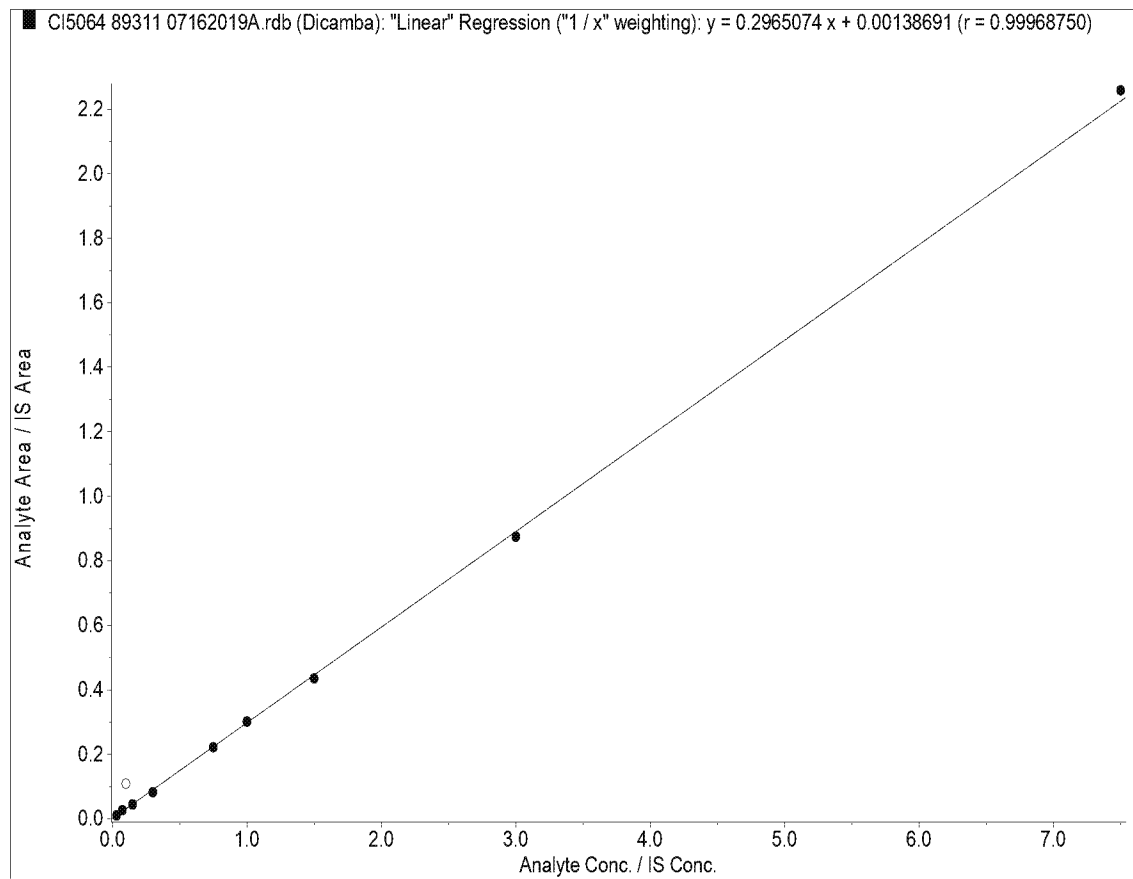


**Representative Chromatogram of 7.50 ng/PUF Standard – Dicamba on PUF**

**EEAL ID: 89311-ST010**



### Representative Calibration Curve – Dicamba on PUF

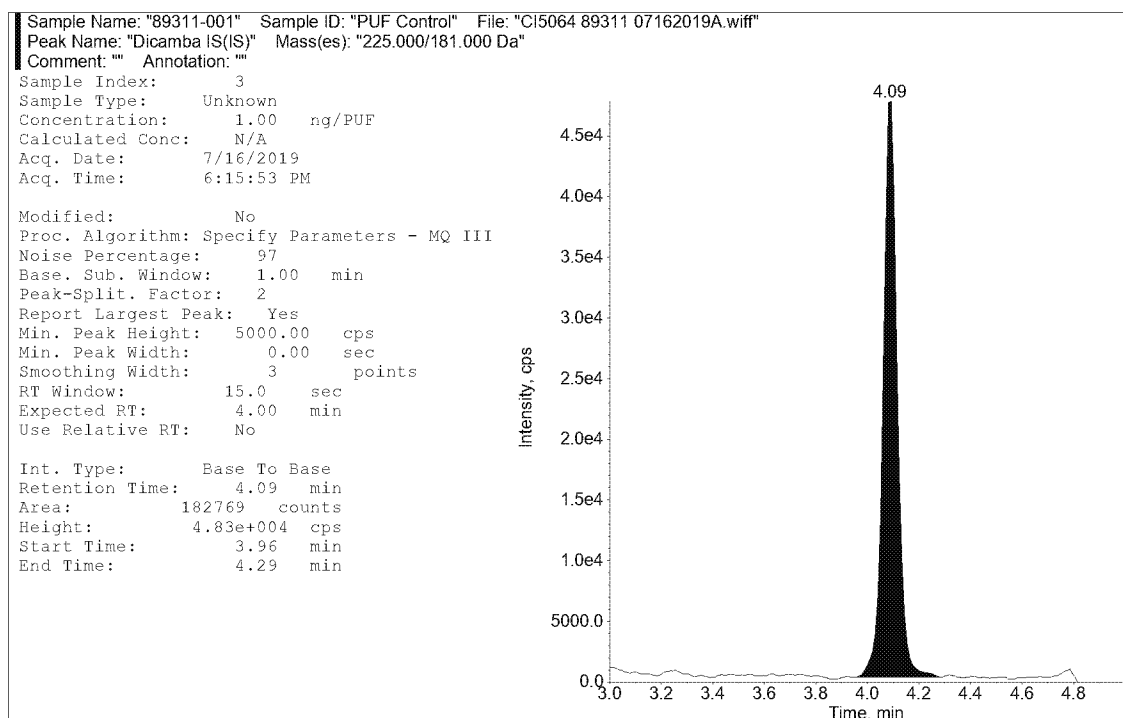
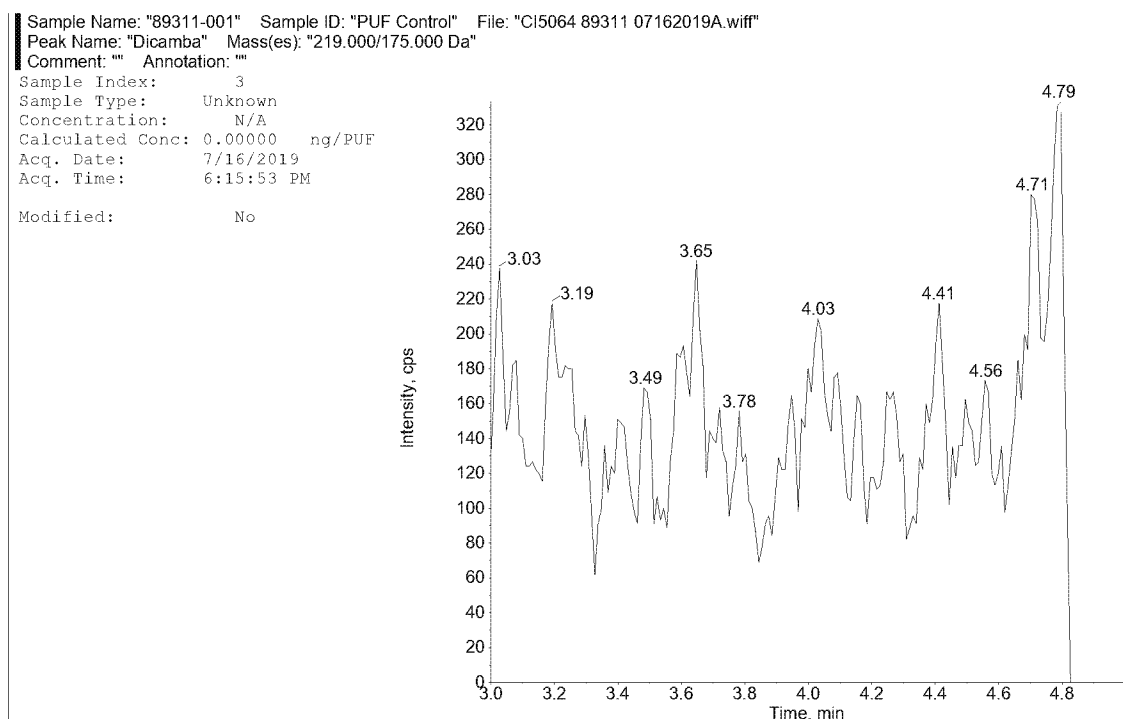


**L.2 Representative Chromatograms of Concurrent Recoveries of Dicamba from Untreated and Fortified PUF Samples**

<b>EEAL ID (89311-)</b>	<b>Sample ID</b>	<b>Sample Comment</b>	<b>Set ID</b>
001	PUF Control	QC Control	001
225	PUF Control	QC Control	005
004	PUF Control + 0.100 ng/PUF	LOQ QC	001
008	PUF Control + 6.00 ng/PUF	Mid QC	
234	PUF Control + 60.0 ng/PUF	High QC	005

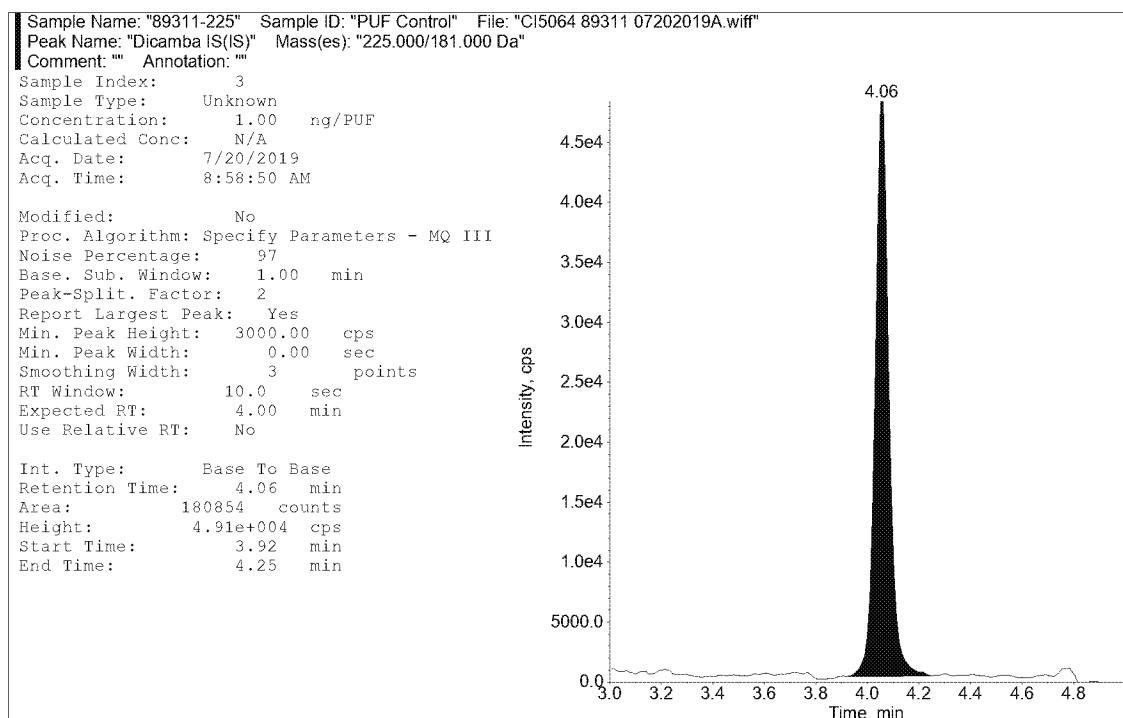
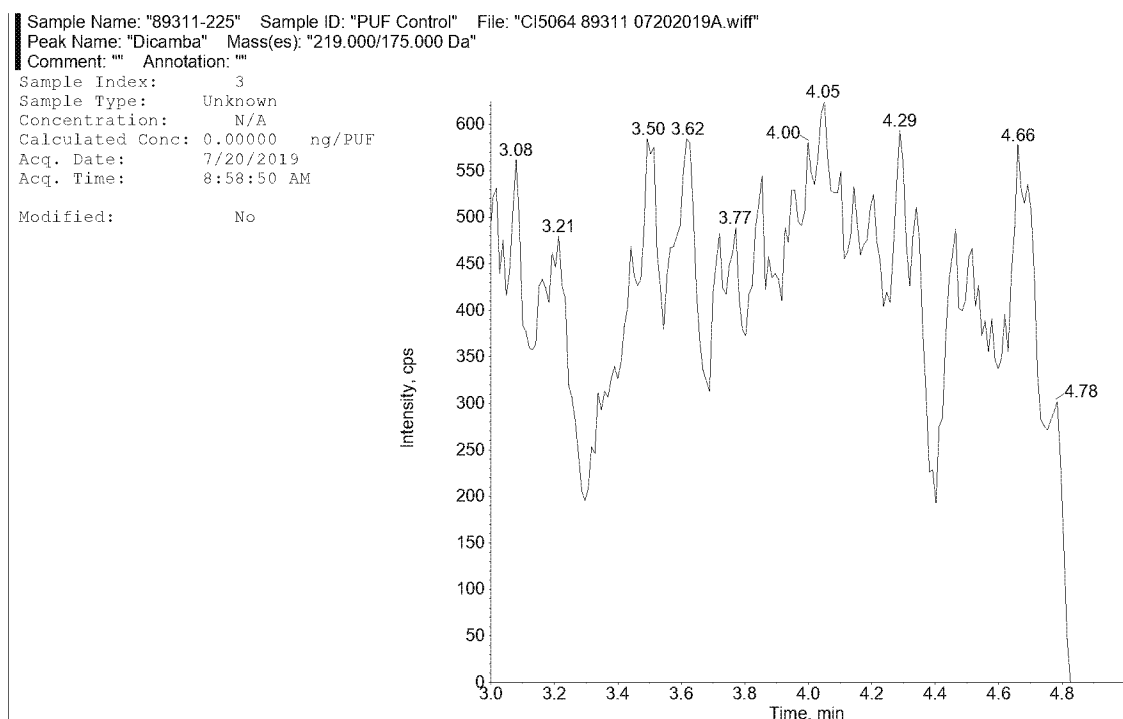
## Representative Chromatogram of Untreated Control PUF

**EEAL ID: 89311-001; Expected Retention Time: 4.09 min.**



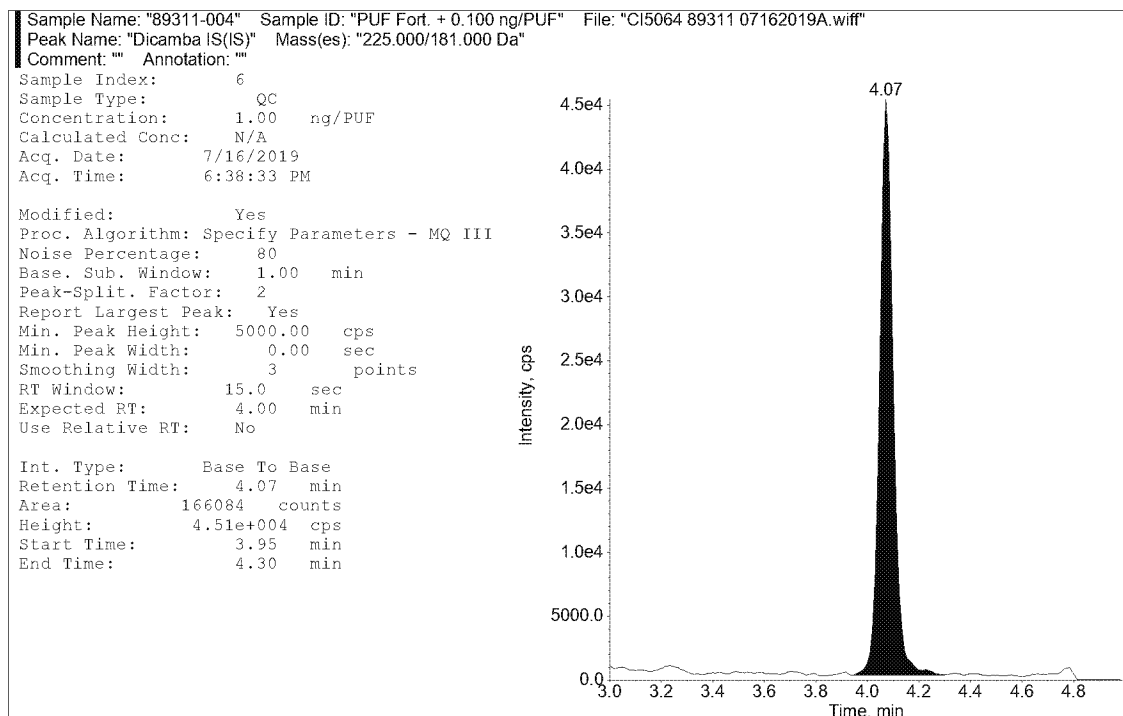
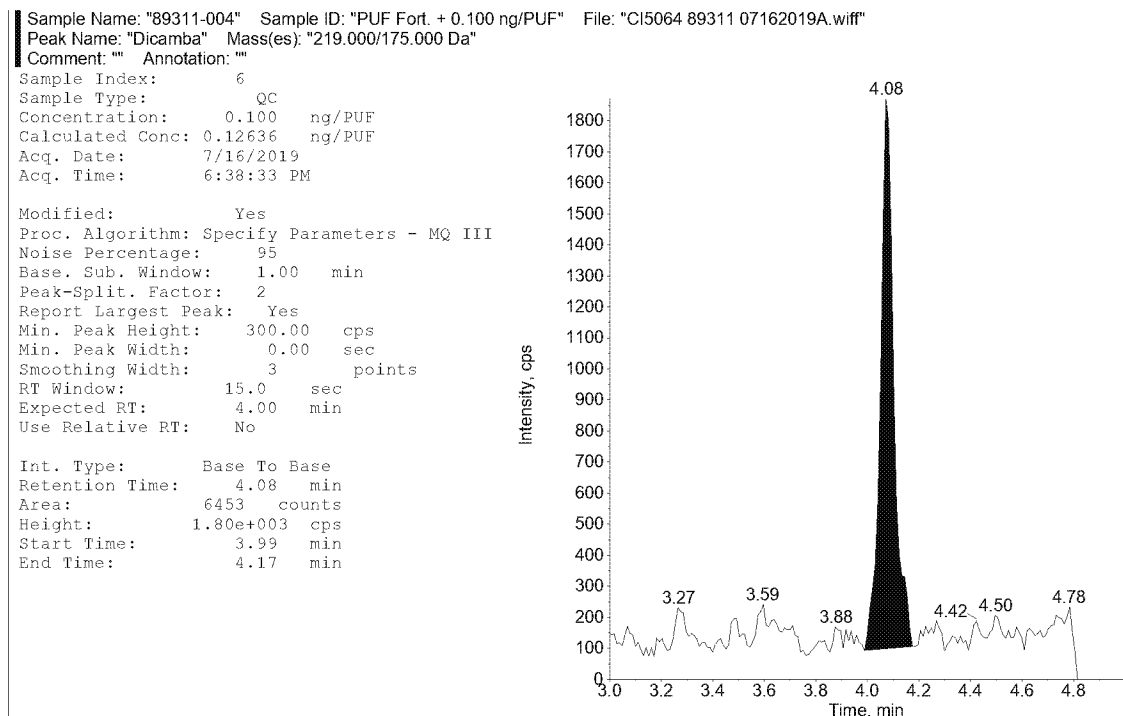
## Representative Chromatogram of Untreated Control PUF (cont'd)

**EEAL ID: 89311-225; Expected Retention Time: 4.06 min.**



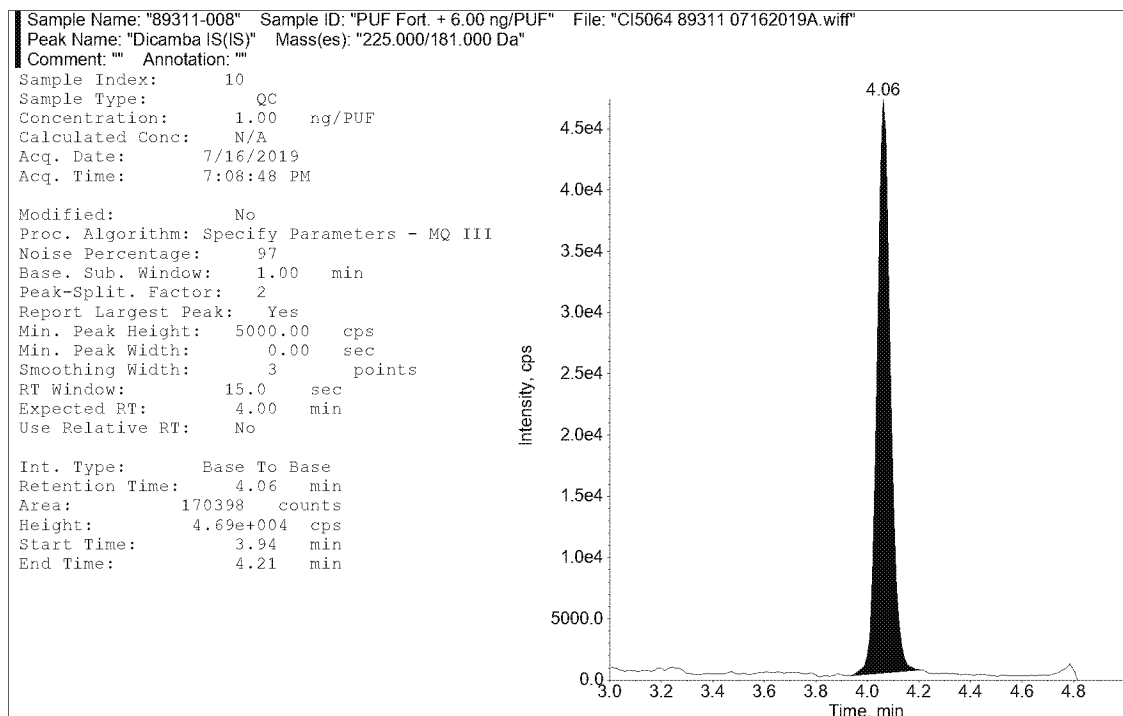
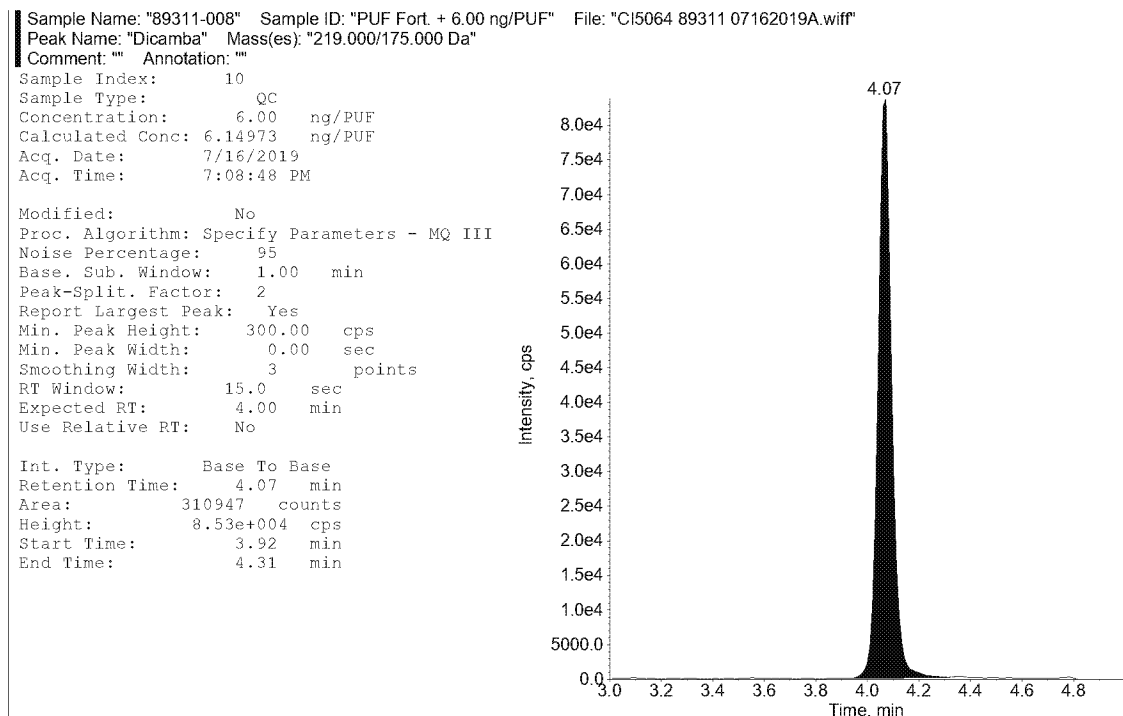
**Representative Chromatogram of Untreated Control Fortified at 0.100 ng/PUF**

**EEAL ID: 89311-004; Recovery: 116% (corrected for control)**



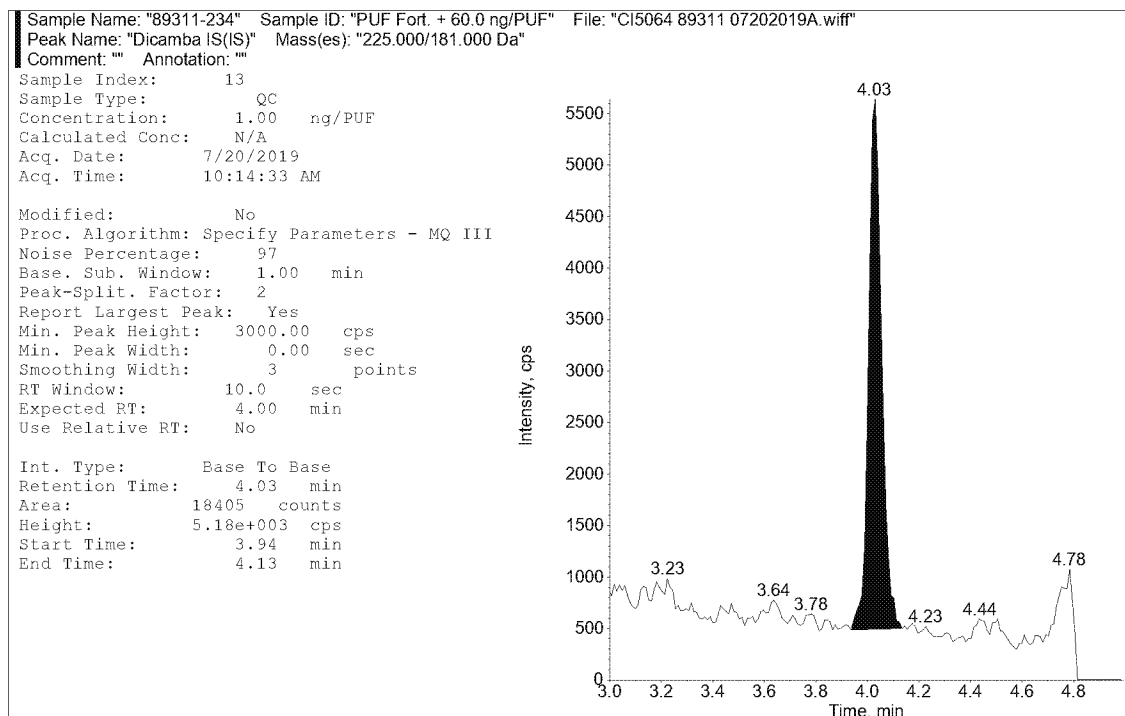
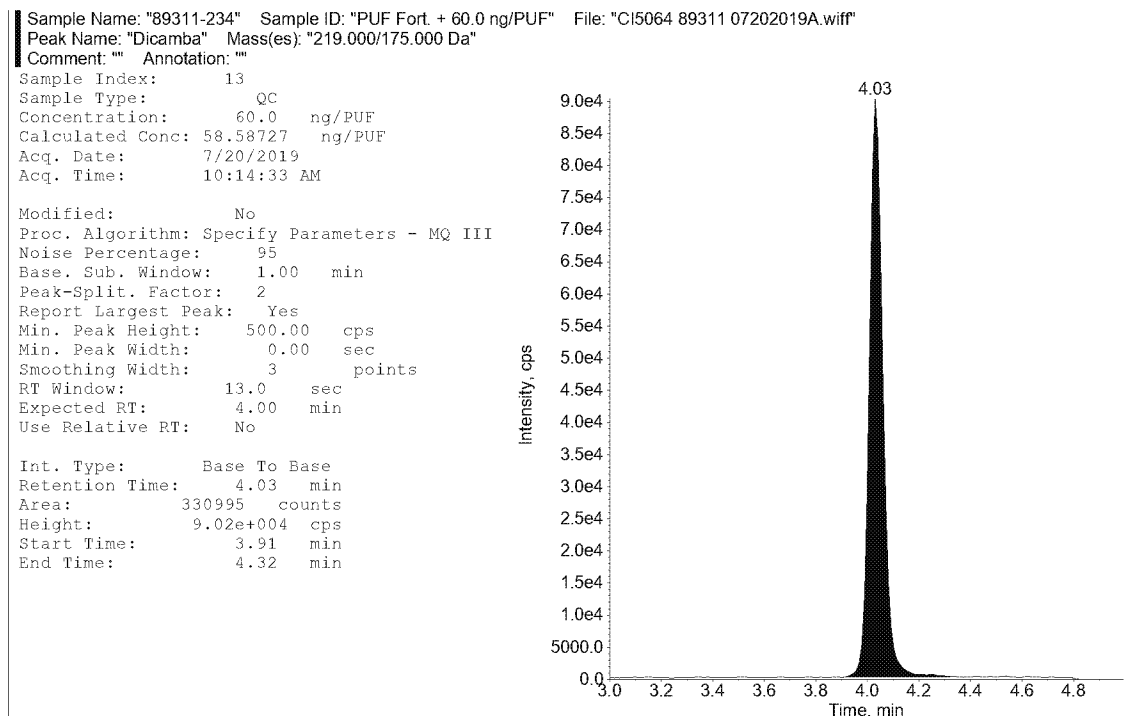
**Representative Chromatogram of Untreated Control Fortified at 6.00 ng/PUF**

**EEAL ID: 89311-008; Recovery: 102%**



**Representative Chromatogram of Untreated Control Fortified at 60.0 ng/PUF**

**EEAL ID: 89311-234; Recovery: 98%**





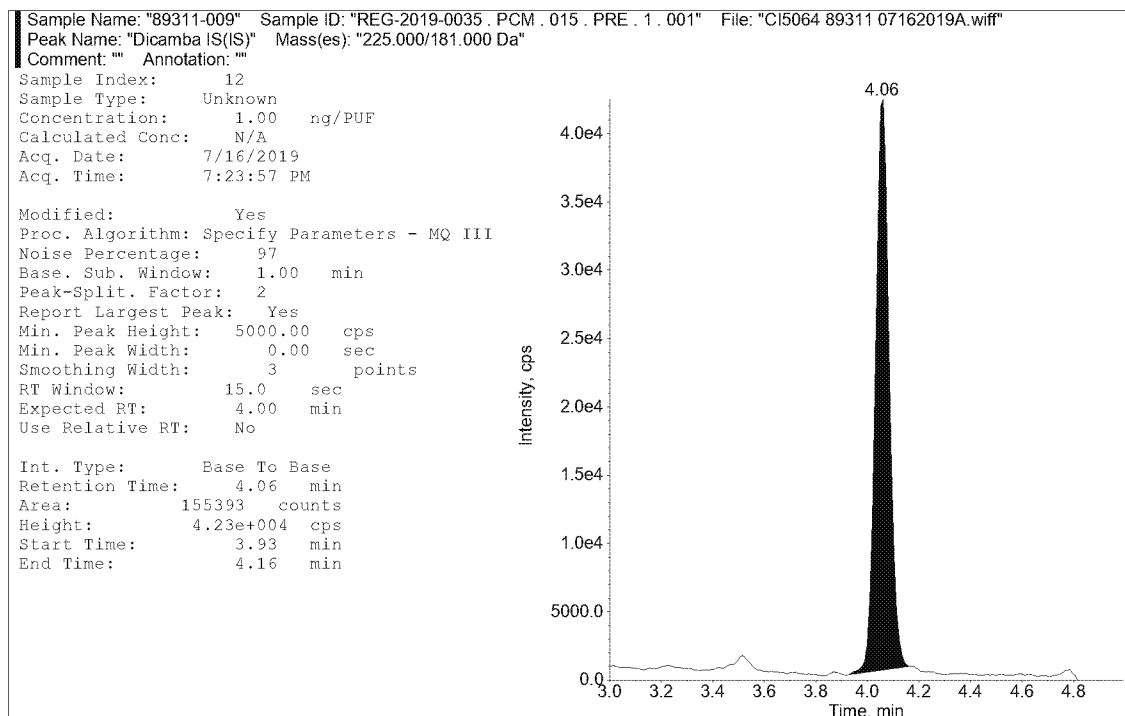
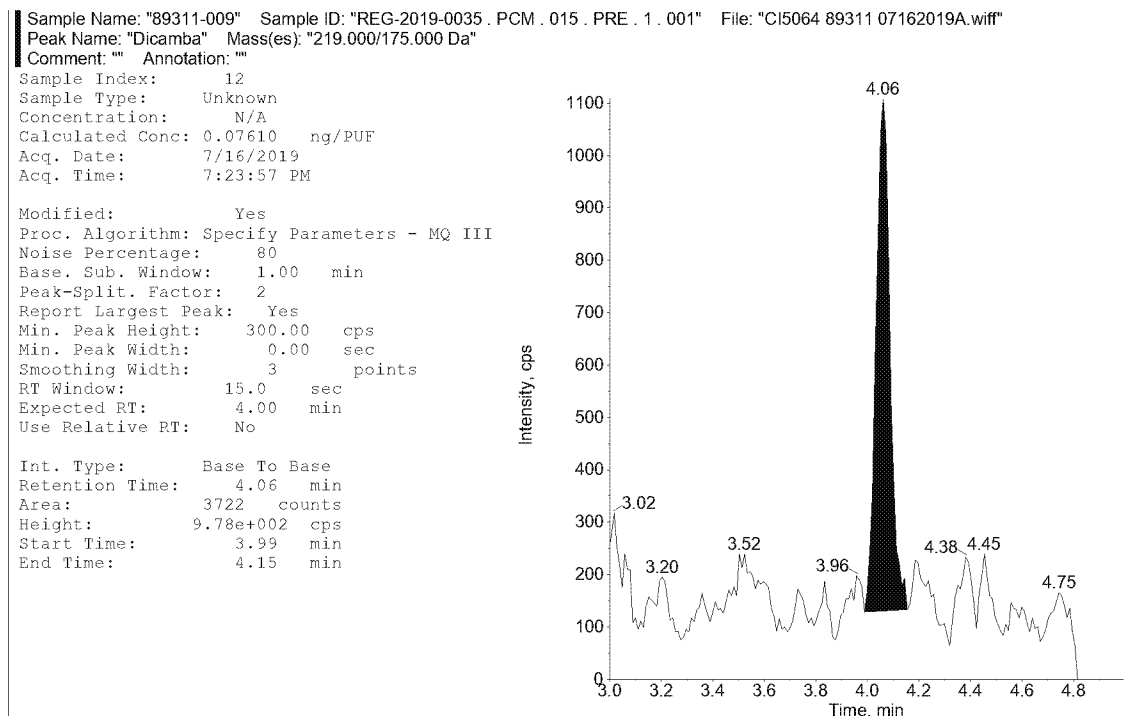
### L.3 Representative Chromatograms of Dicamba PUF Samples

EEAL ID (89311-)	Sample ID	Sample Comment	Set ID
009	REG-2019-0035 . PCM . 015 . PRE . 1 . 001	PUF Center Mast 0.15 m - pre-application	001
041	REG-2019-0035 . PCM . 055 . 12-24 hr . 1 . 055	PUF Center Mast 0.55 m – 12-24 hours	
140	REG-2019-0035 . PCM . 033 . 84-96 hr . 1 . 138	PUF Center Mast 0.33 m – 84-96 hours	003
246	REG-2019-0035 . PCM . 150 . 156-168 hr . 1 . 225	PUF Center Mast 1.50 m – 156-168 hours	005
017	REG-2019-0035 . POF . A . 0-6 hr . 1 . 031	PUF Perimeter A – 0-6 hours	001
047	REG-2019-0035 . POF . C . 12-24 hr . 1 . 061	PUF Perimeter C – 12-24 hours	
152	REG-2019-0035 . POF . H . 84-96 hr . 1 . 150	PUF Perimeter H – 84-96 hours	003
251	REG-2019-0035 . POF . D . 156-168 hr . 1 . 230	PUF Perimeter D – 156-168 hours	005

## Representative Chromatogram of Dicamba PUF

EEAL ID: 89311-009

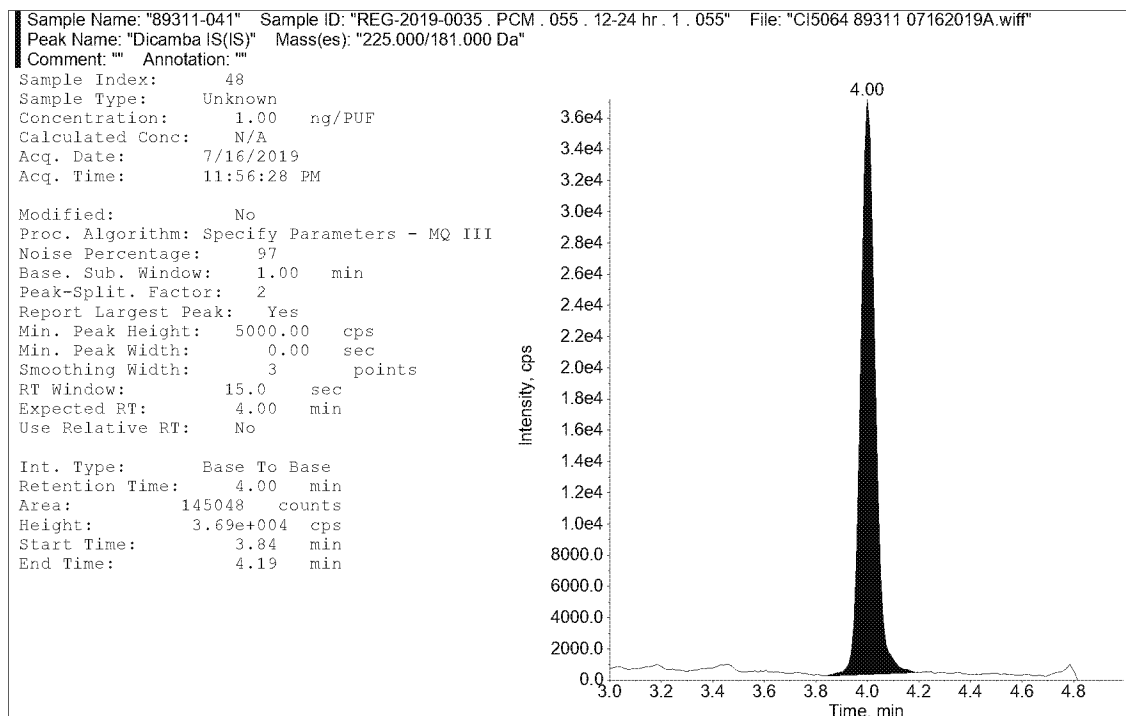
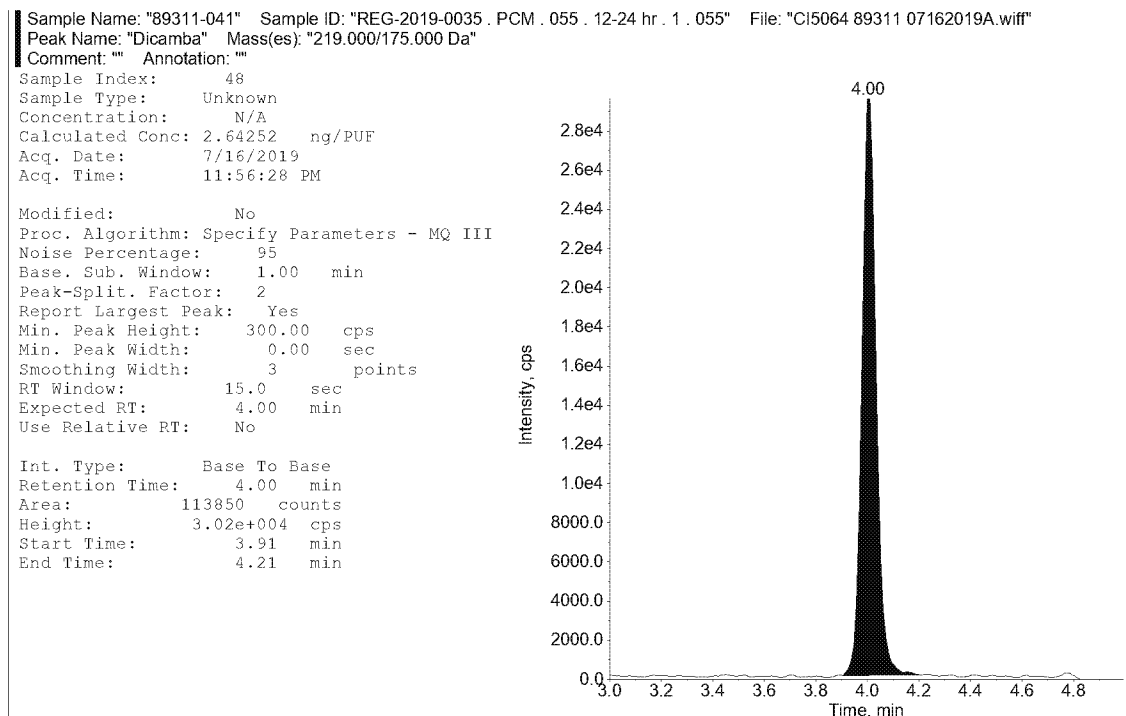
Sample ID: REG-2019-0035 . PCM . 015 . PRE . 1 . 001



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-041**

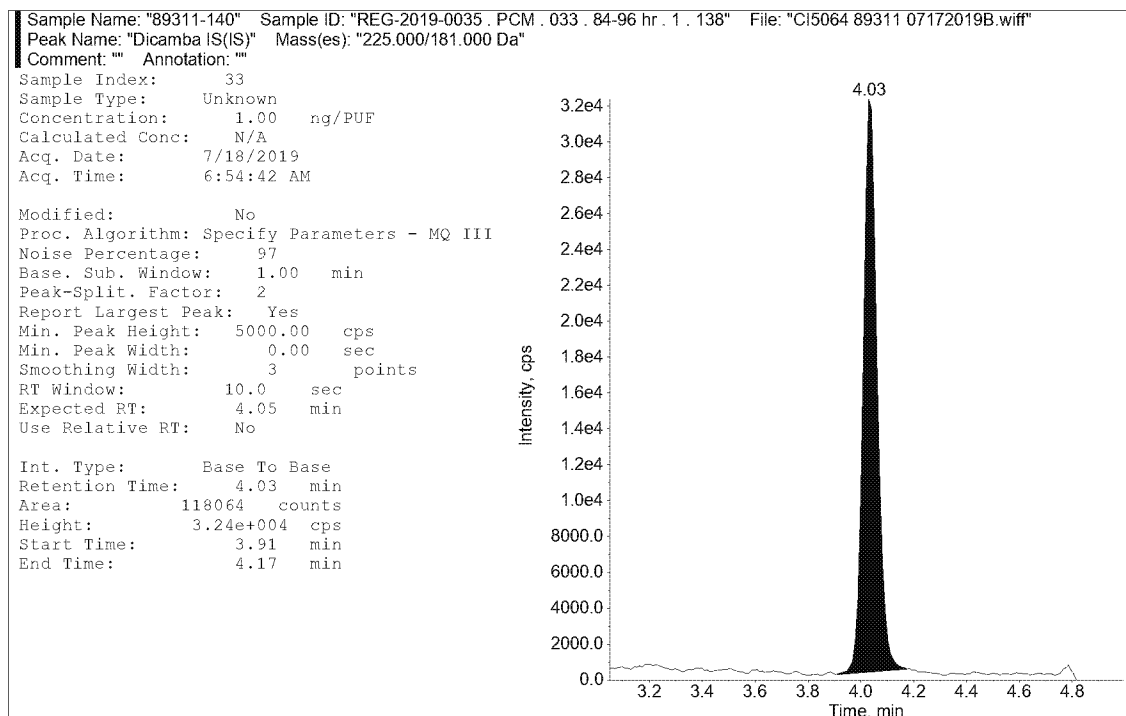
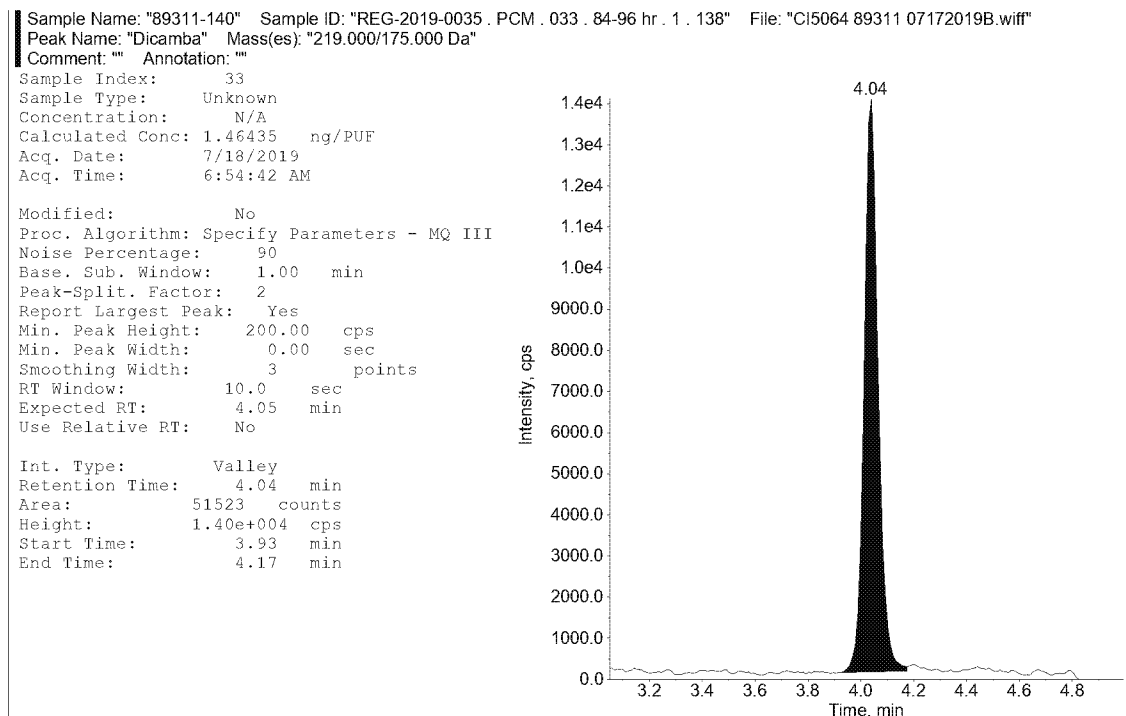
**Sample ID: REG-2019-0035 . PCM . 055 . 12-24 hr . 1 . 055**



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-140**

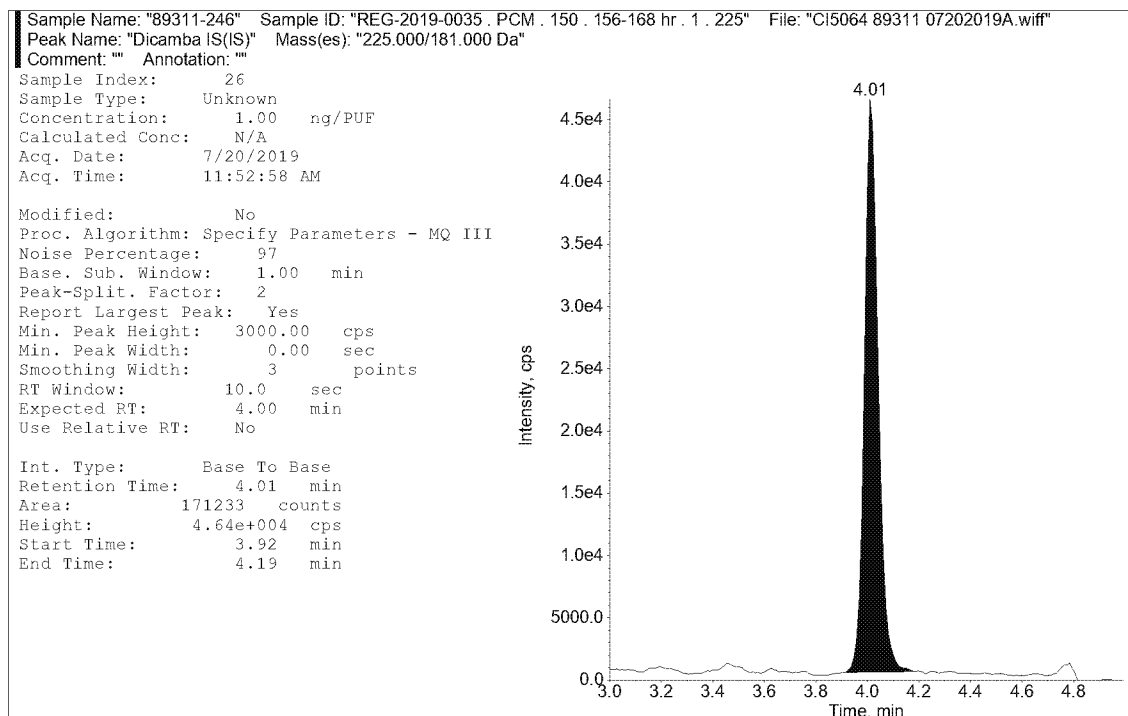
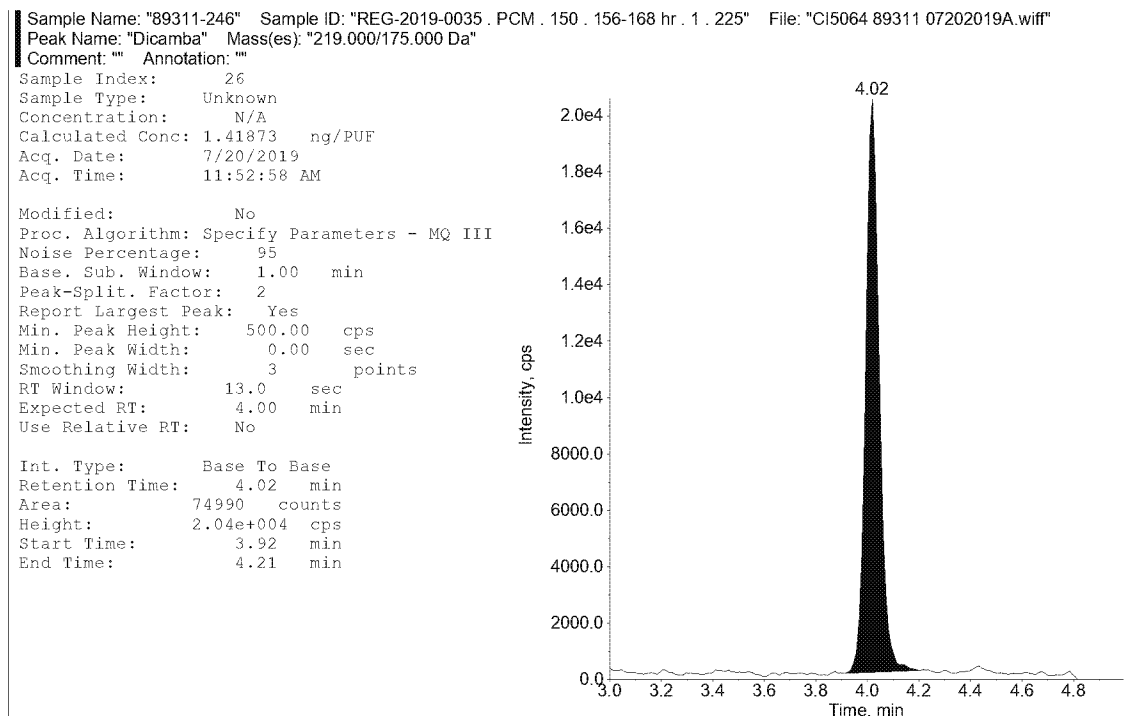
**Sample ID: REG-2019-0035 . PCM . 033 . 84-96 hr . 1 . 138**



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-246**

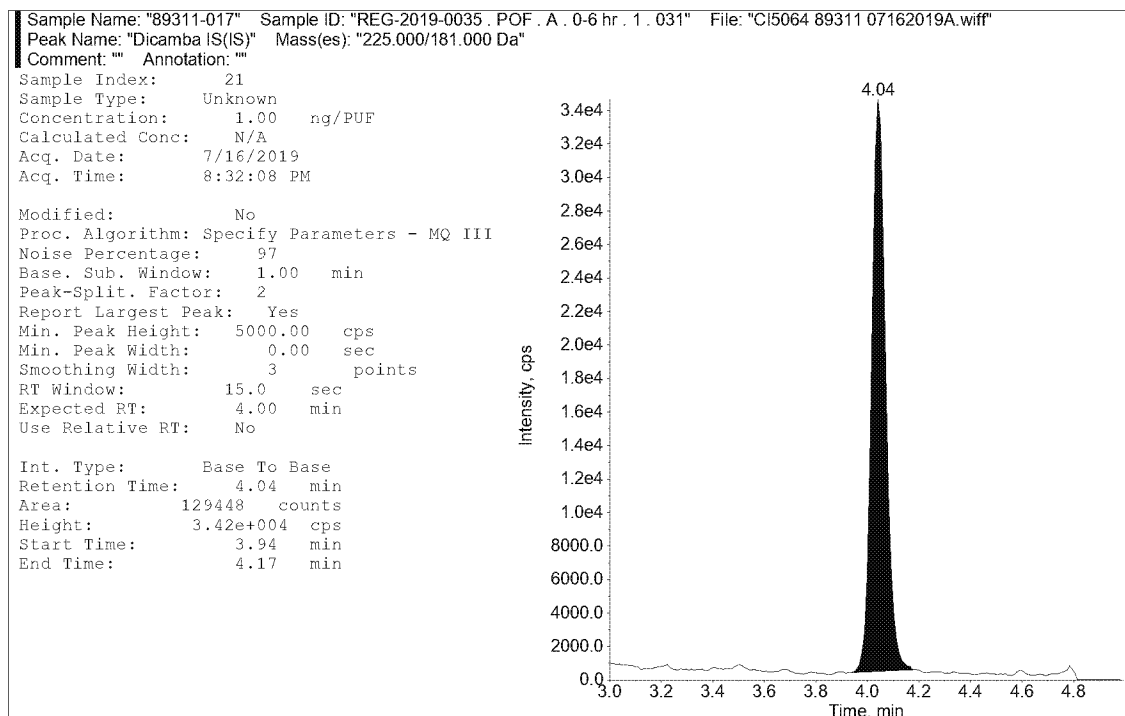
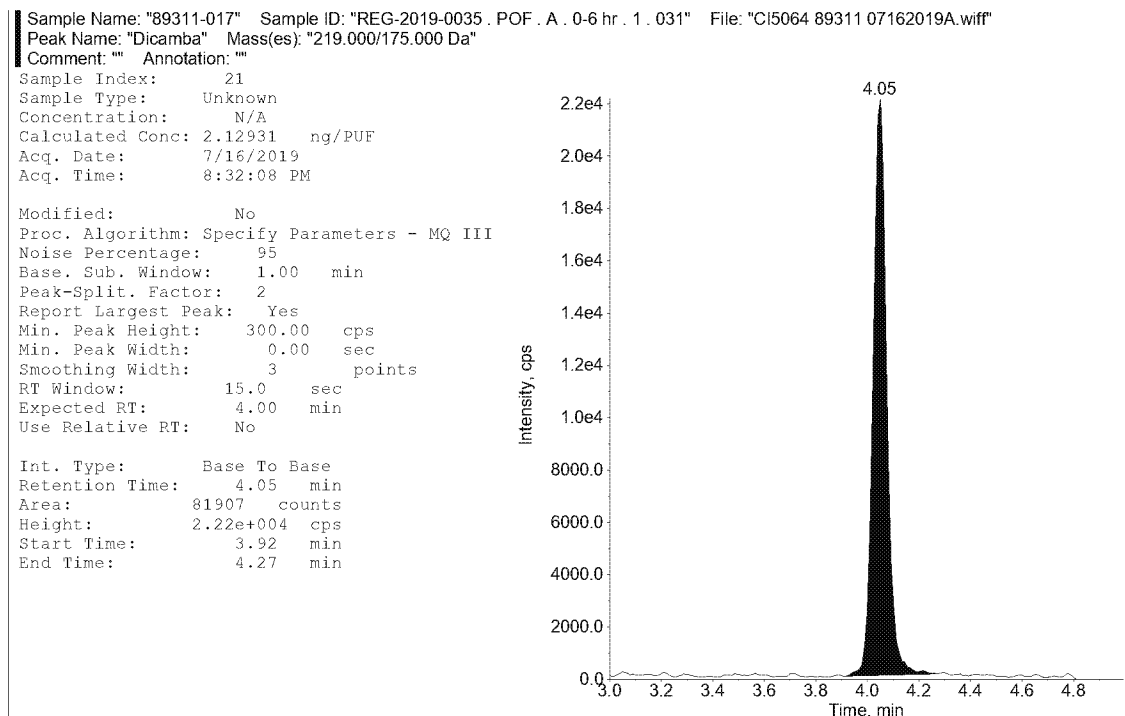
**Sample ID: REG-2019-0035 . PCM . 150 . 156-168 hr . 1 . 225**



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-017**

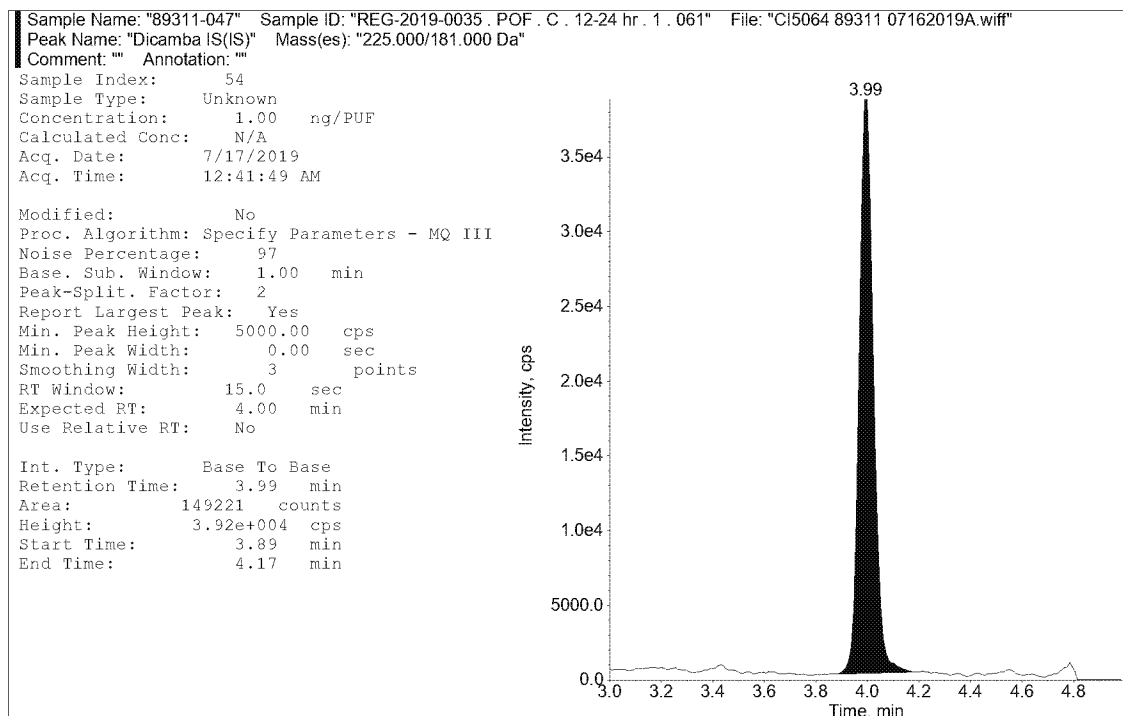
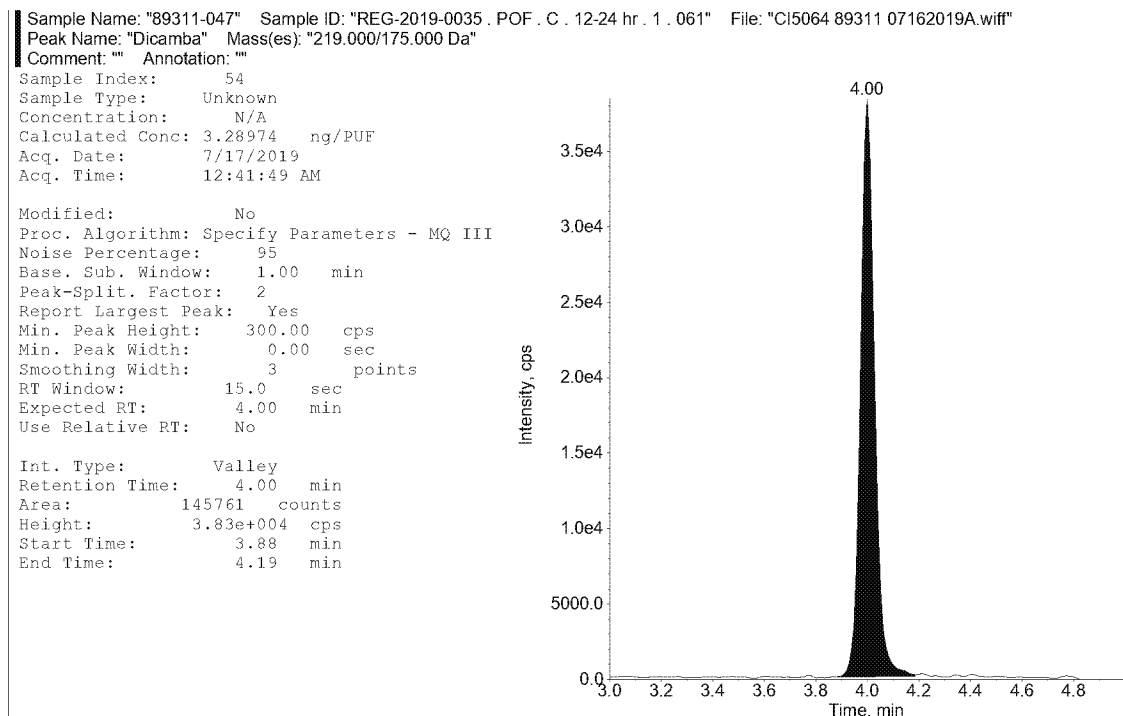
**Sample ID: REG-2019-0035 . POF . A . 0-6 hr . 1 . 031**



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-047**

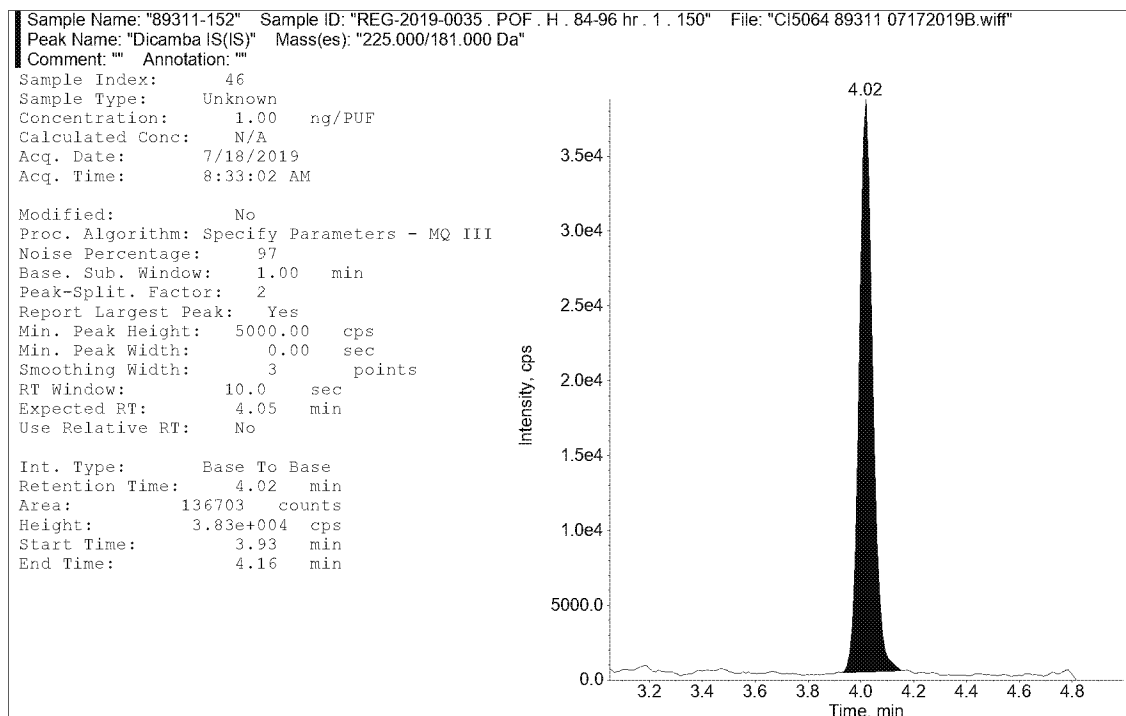
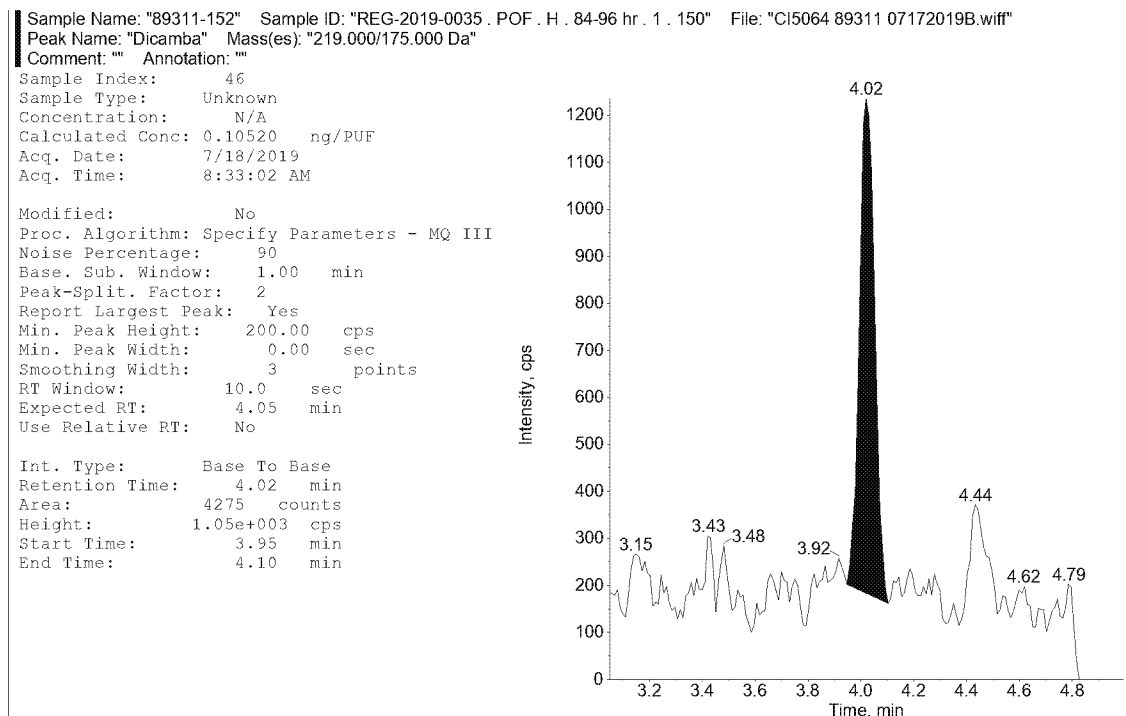
**Sample ID: REG-2019-0035 . POF . C . 12-24 hr . 1 . 061**



**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-152**

**Sample ID: REG-2019-0035 . POF . H . 84-96 hr . 1 . 150**

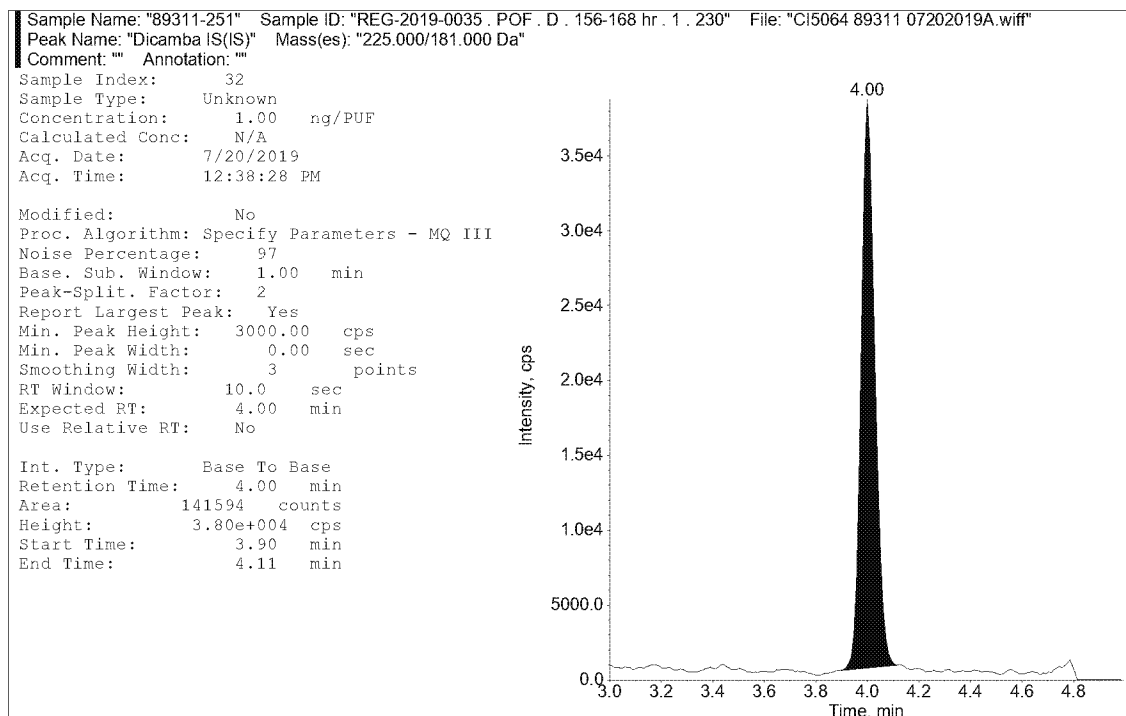
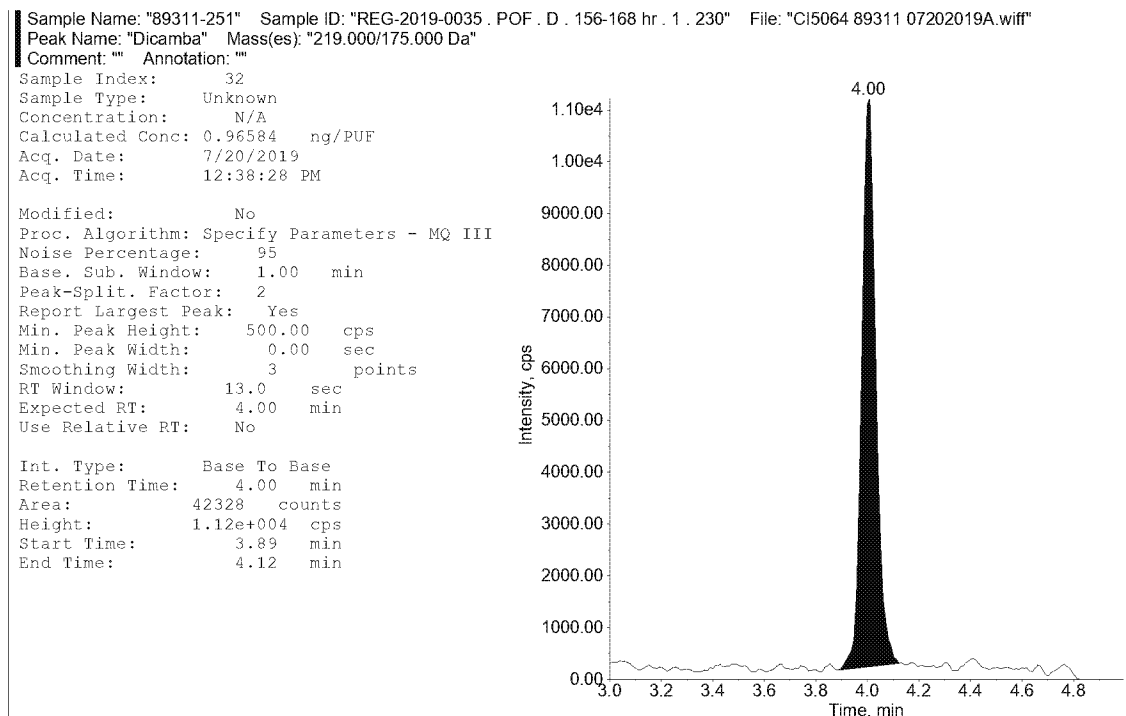




**Representative Chromatogram of Dicamba PUF (cont'd)**

**EEAL ID: 89311-251**

**Sample ID: REG-2019-0035 . POF . D . 156-168 hr . 1 . 230**



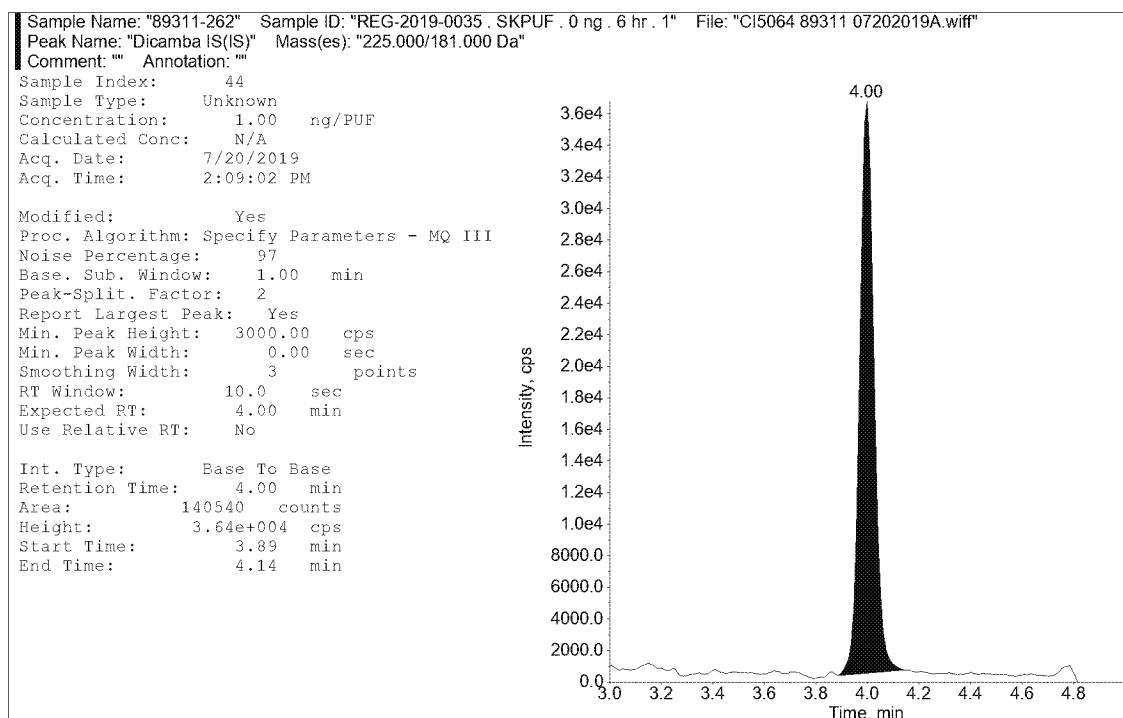
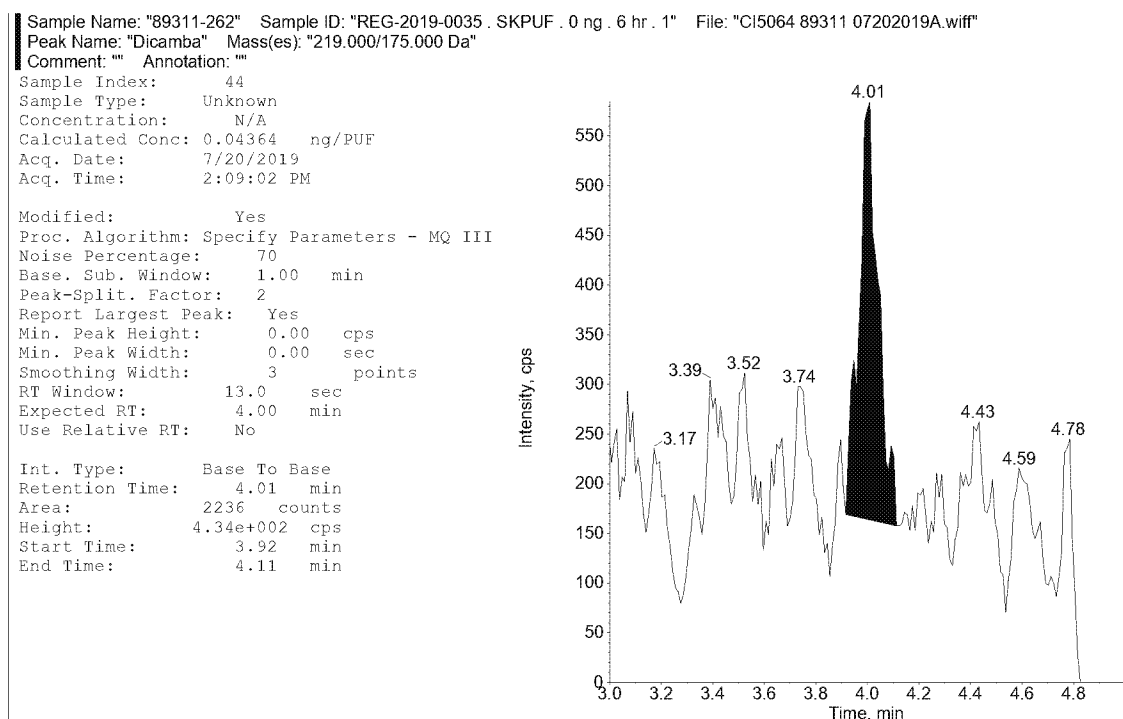
#### L.4 Representative Chromatograms of Field Exposed Spike and Transit Stability PUF Samples

EEAL ID (89311-)	Sample ID	Sample Comment	Set ID
262	REG-2019-0035 . SKPUF . 0 ng . 6 hr . 1	Untreated Control – 6 hours	005
266	REG-2019-0035 . SKPUF . 3 ng . 6 hr . 2	Field Spike, 6 hours, 3 ng/PUF	
270	REG-2019-0035 . SKPUF . 10 ng . 6 hr . 3	Field Spike, 6 hours, 10 ng/PUF	
273	REG-2019-0035 . SKPUF . 30 ng . 6 hr . 3	Field Spike, 6 hours, 30 ng/PUF	
274	REG-2019-0035 . SKPUF . 0 ng . 12 hr . 1	Untreated Control – 12 hours	
277	REG-2019-0035 . SKPUF . 3 ng . 12 hr . 1	Field Spike, 12 hours, 3 ng/PUF	
282	REG-2019-0035 . SKPUF . 10 ng . 12 hr . 3	Field Spike, 12 hours, 10 ng/PUF	
284	REG-2019-0035 . SKPUF . 30 ng . 12 hr . 2	Field Spike, 12 hours, 30 ng/PUF	
256	REG-2019-0035 . TSPUF . 0 ng . 1	Untreated Control – Transit	
259	REG-2019-0035 . TSPUF . 30 ng . 1	Transit, 30 ng/PUF	

## Representative Chromatograms from Field Exposed PUF

EEAL ID: 89311-262

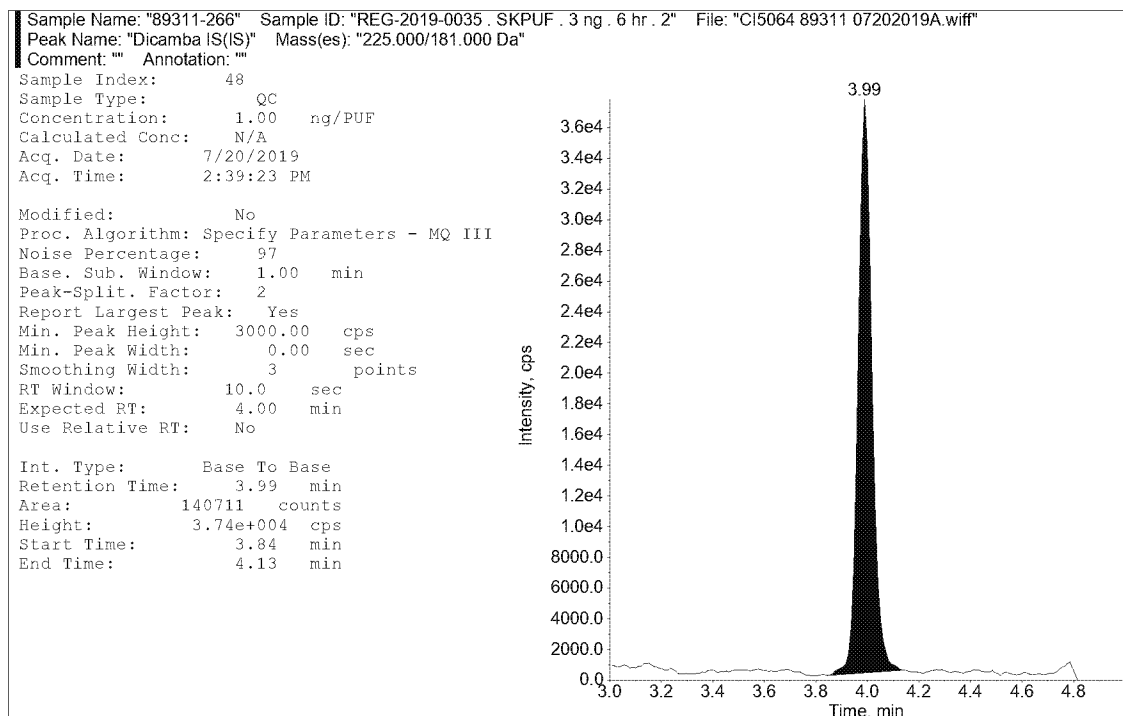
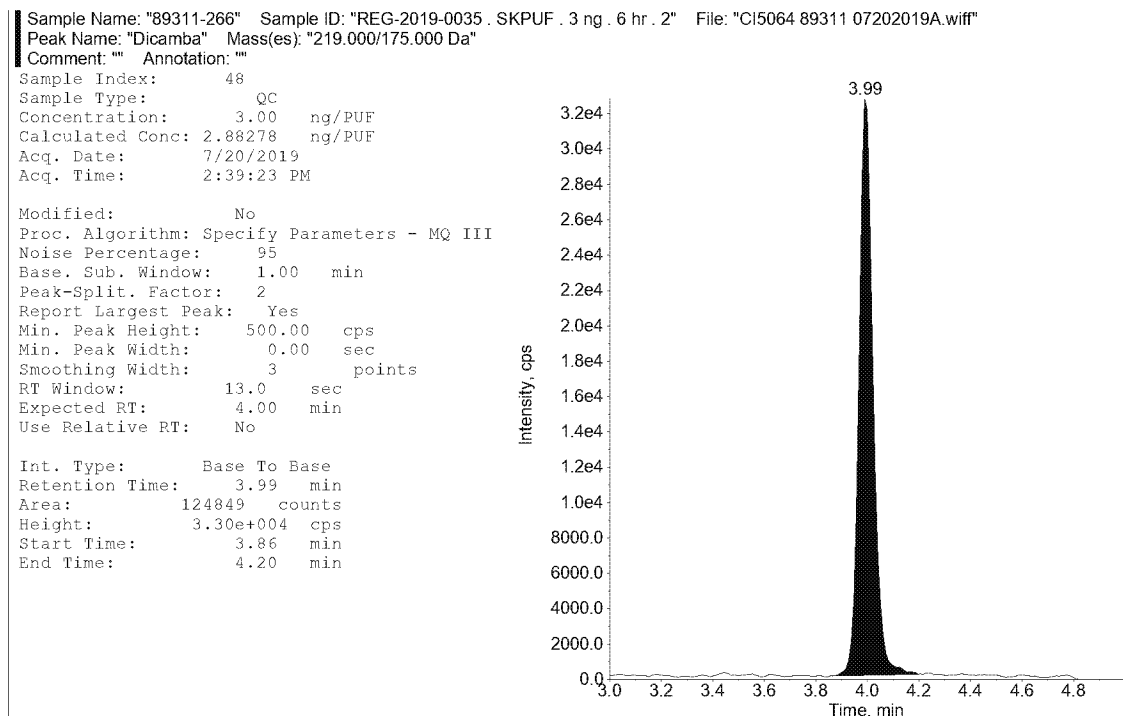
Sample ID: REG-2019-0035 . SKPUF . 0 ng . 6 hr . 1



Representative Chromatograms from Field Exposed PUF (cont'd)

EEAL ID: 89311-266

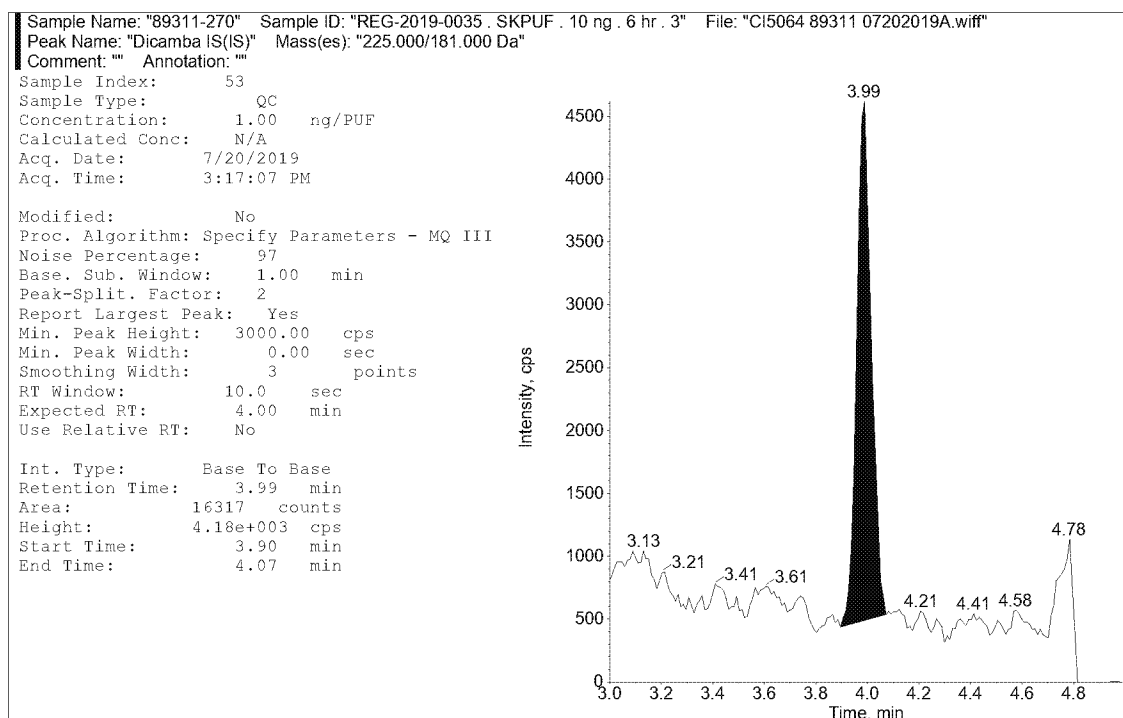
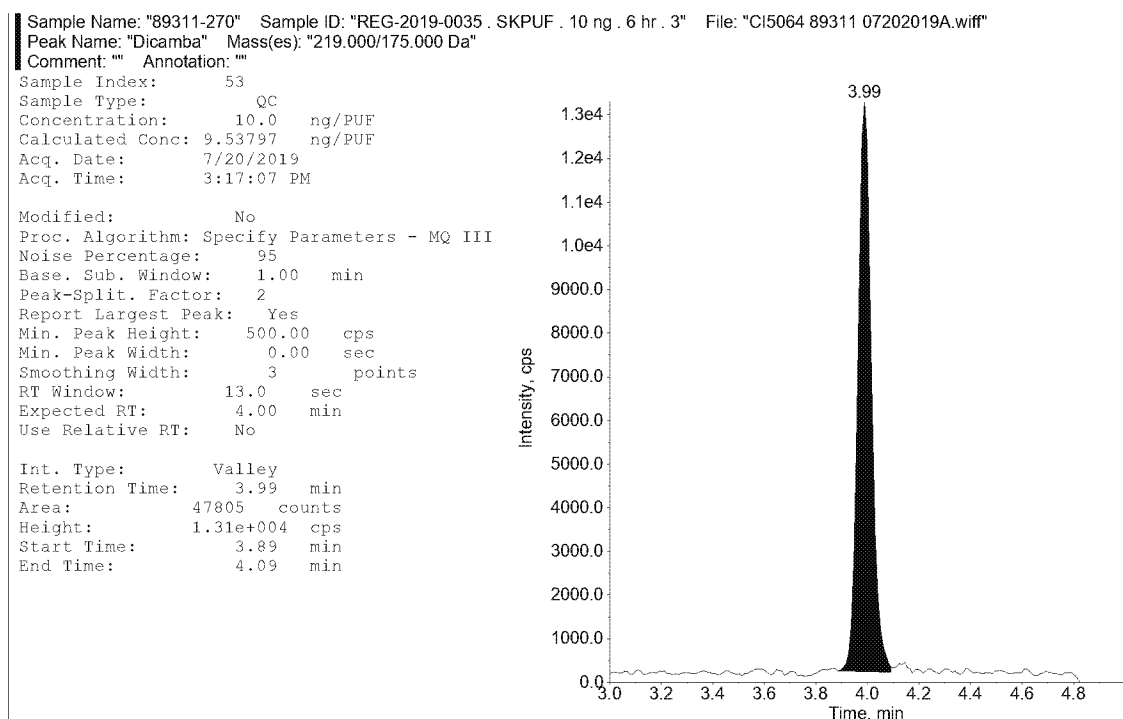
Sample ID: REG-2019-0035 . SKPUF . 3 ng . 6 hr . 2



**Representative Chromatograms from Field Exposed PUF (cont'd)**

**EEAL ID: 89311-270**

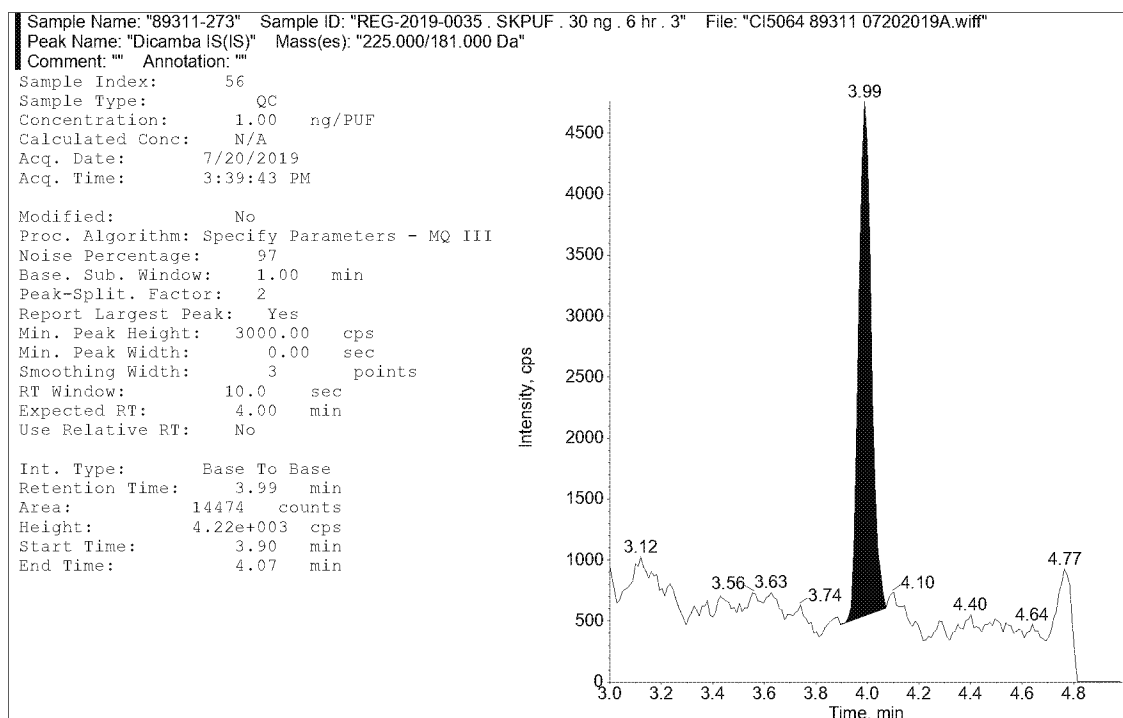
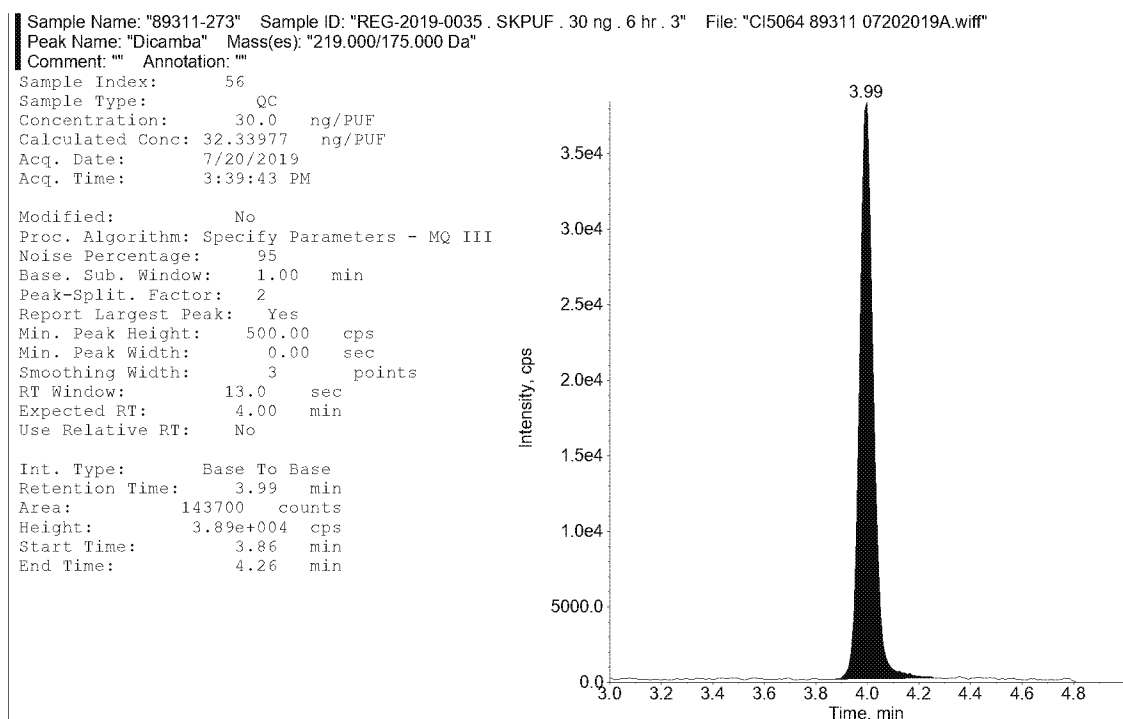
**Sample ID: REG-2019-0035 . SKPUF . 10 ng . 6 hr . 3**



**Representative Chromatograms from Field Exposed PUF (cont'd)**

**EEAL ID: 89311-273**

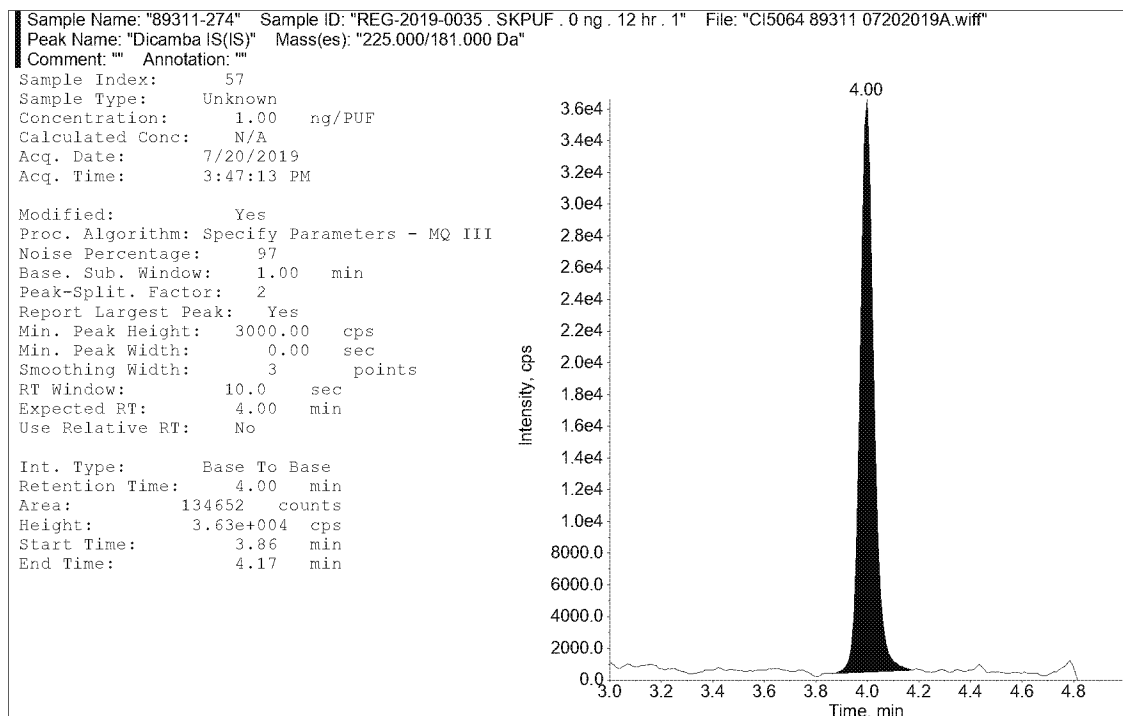
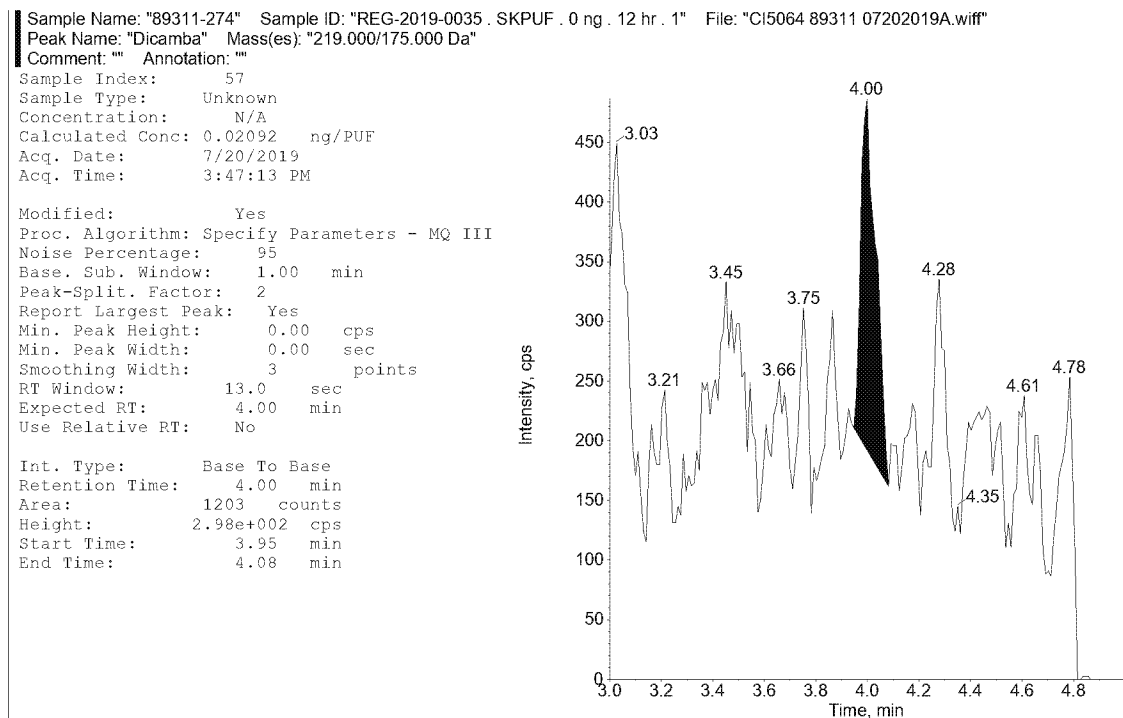
**Sample ID: REG-2019-0035 . SKPUF . 30 ng . 6 hr . 3**



Representative Chromatograms from Field Exposed PUF (cont'd)

EEAL ID: 89311-274

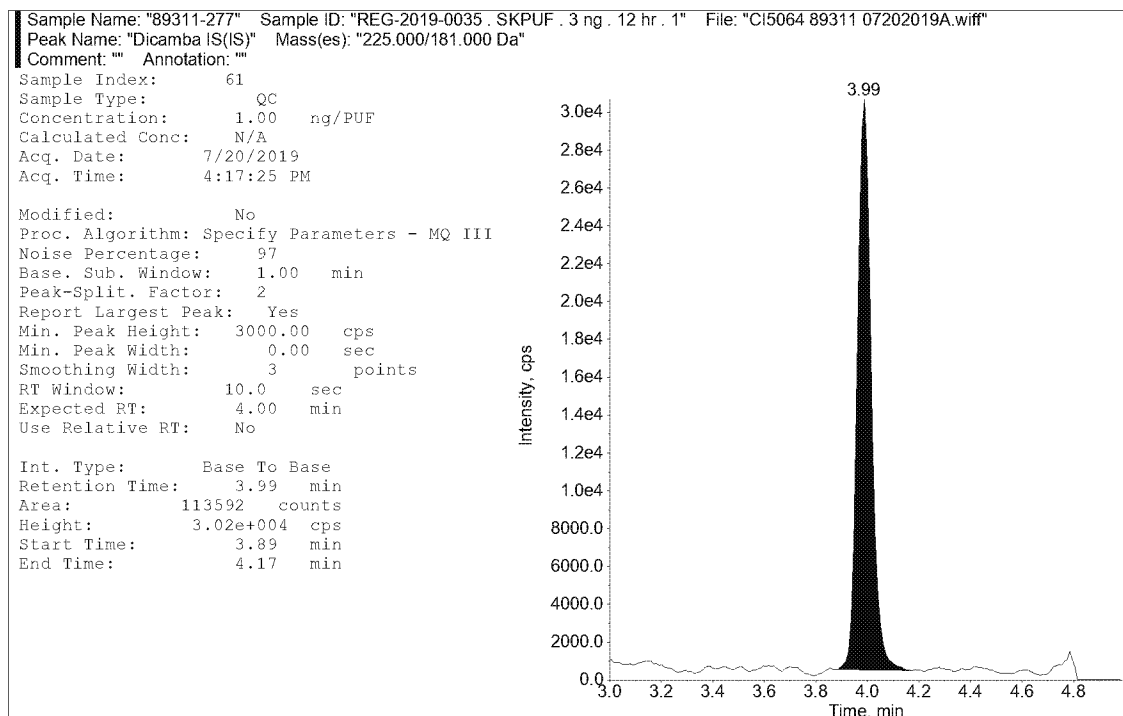
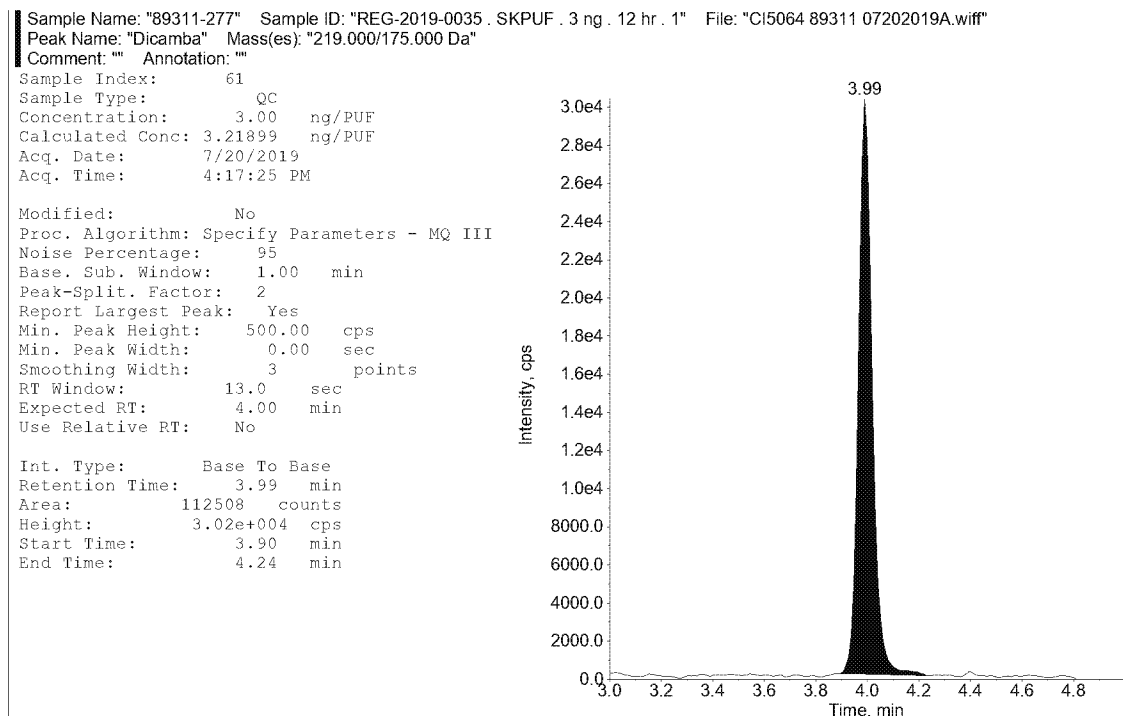
Sample ID: REG-2019-0035 . SKPUF . 0 ng . 12 hr . 1



**Representative Chromatograms from Field Exposed PUF (cont'd)**

**EEAL ID: 89311-277**

**Sample ID: REG-2019-0035 . SKPUF . 3 ng . 12 hr . 1**

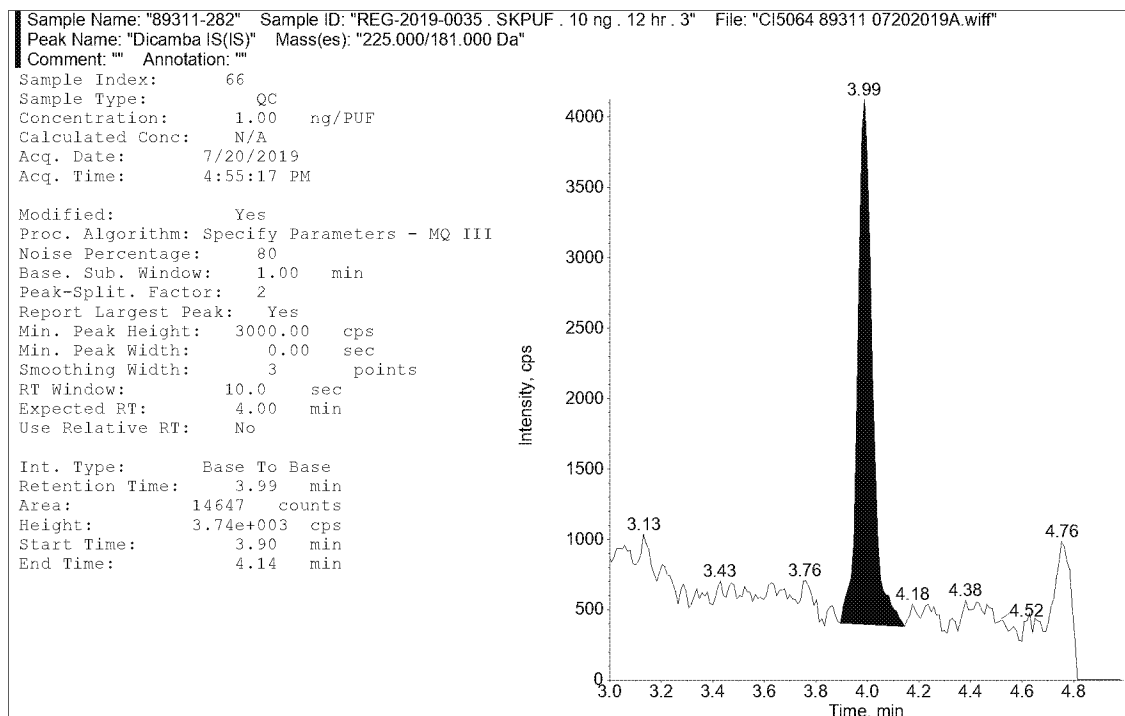
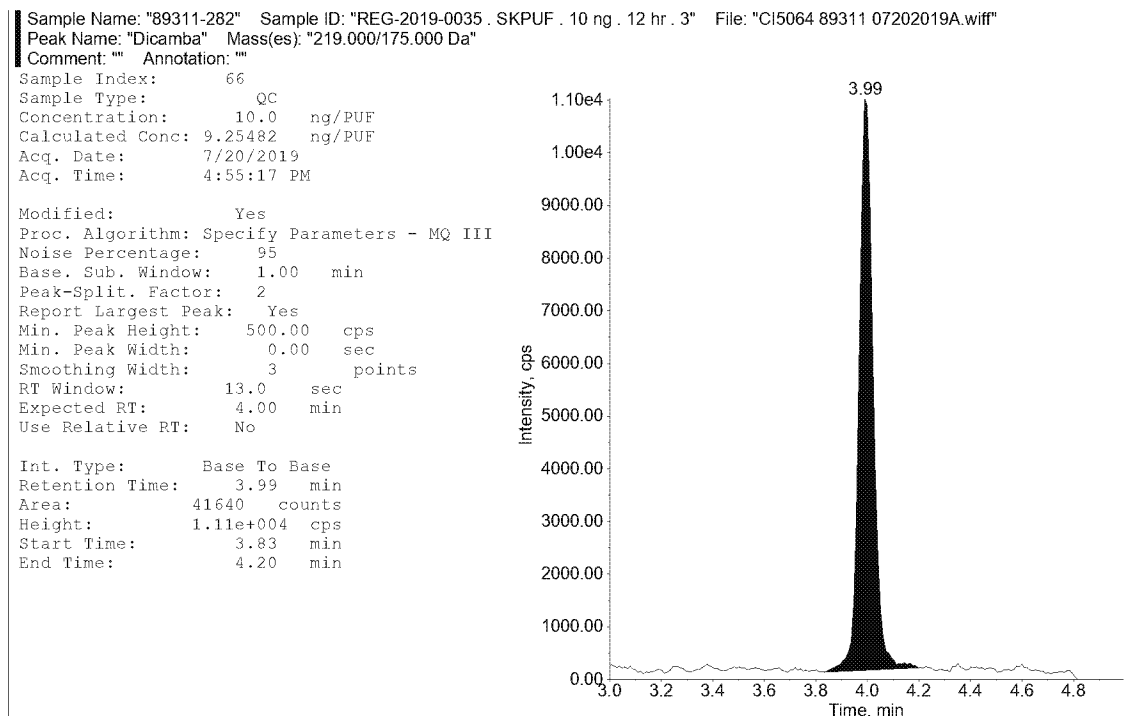




**Representative Chromatograms from Field Exposed PUF (cont'd)**

**EEAL ID: 89311-282**

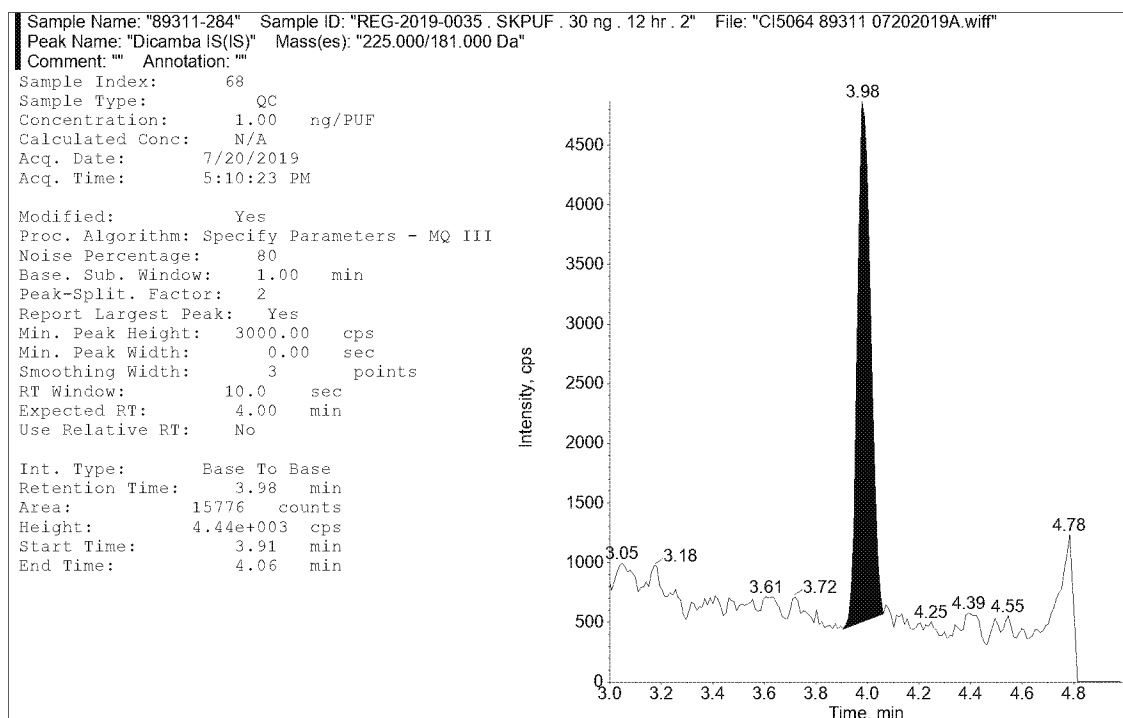
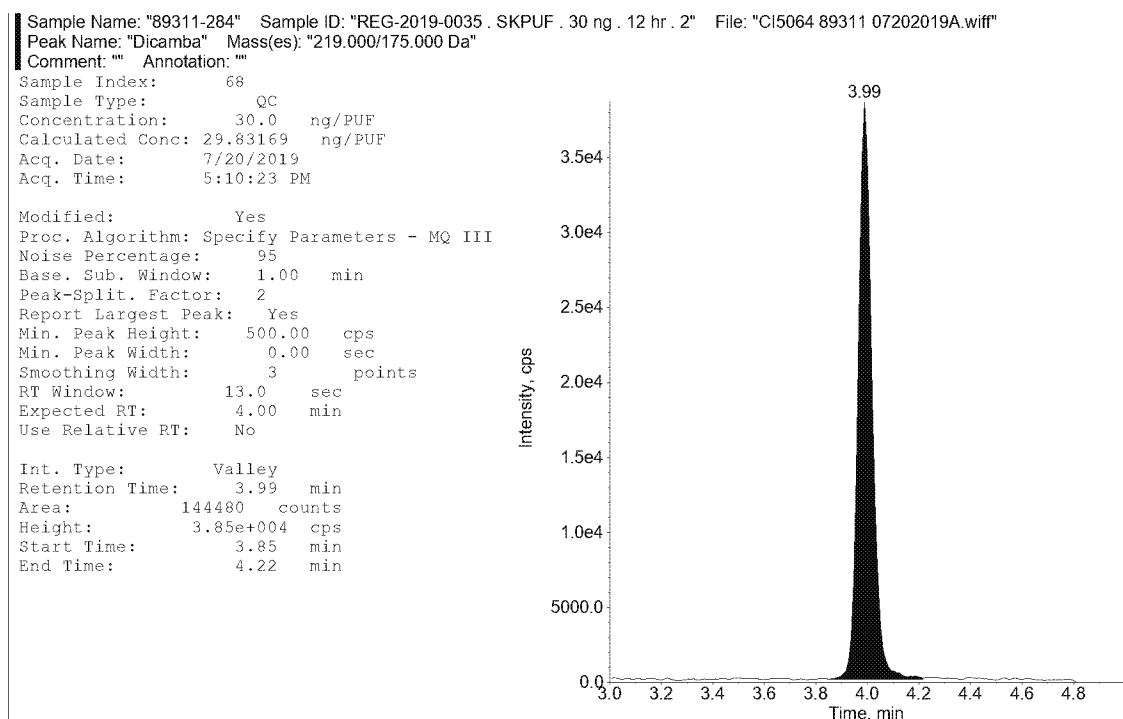
**Sample ID: REG-2019-0035 . SKPUF . 10 ng . 12 hr . 3**



**Representative Chromatograms from Field Exposed PUF (cont'd)**

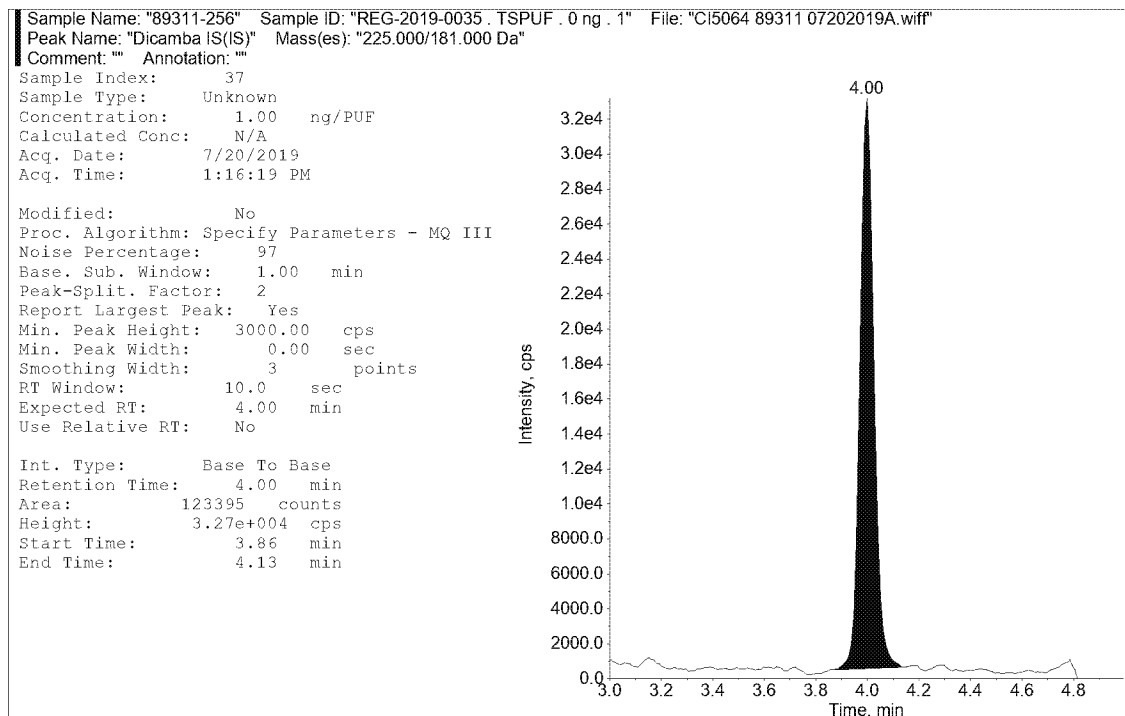
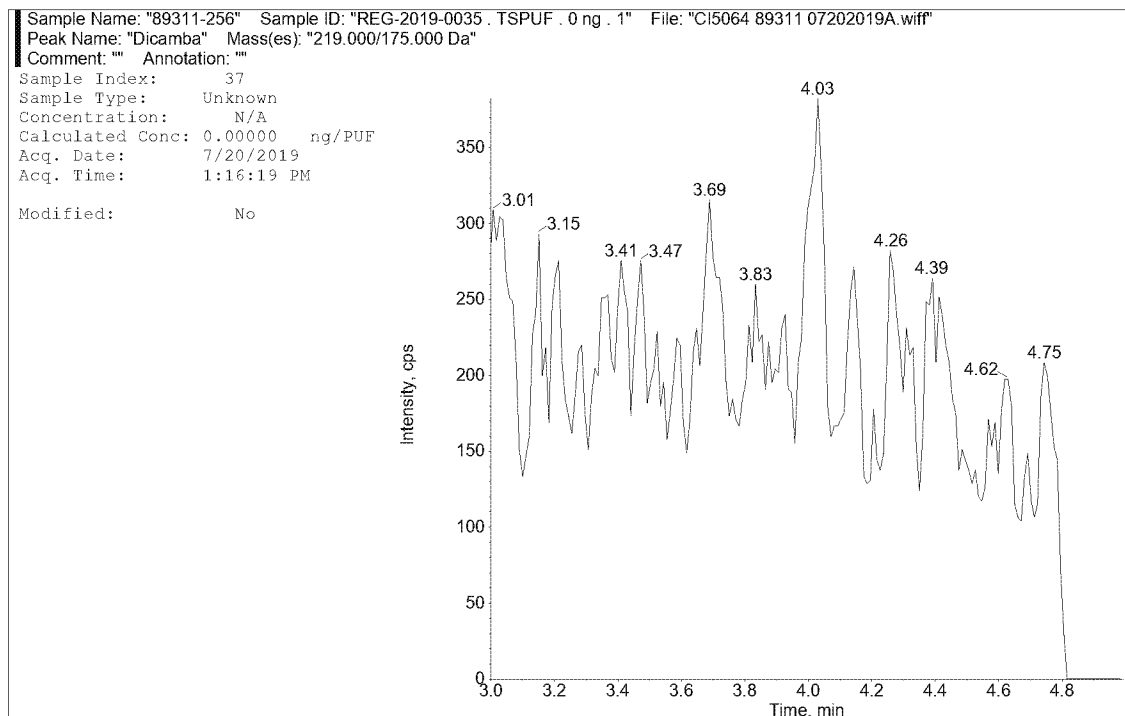
**EEAL ID: 89311-284**

**Sample ID: REG-2019-0035 . SKPUF . 30 ng . 12 hr . 2**



## Representative Chromatograms from Transit Stability PUF

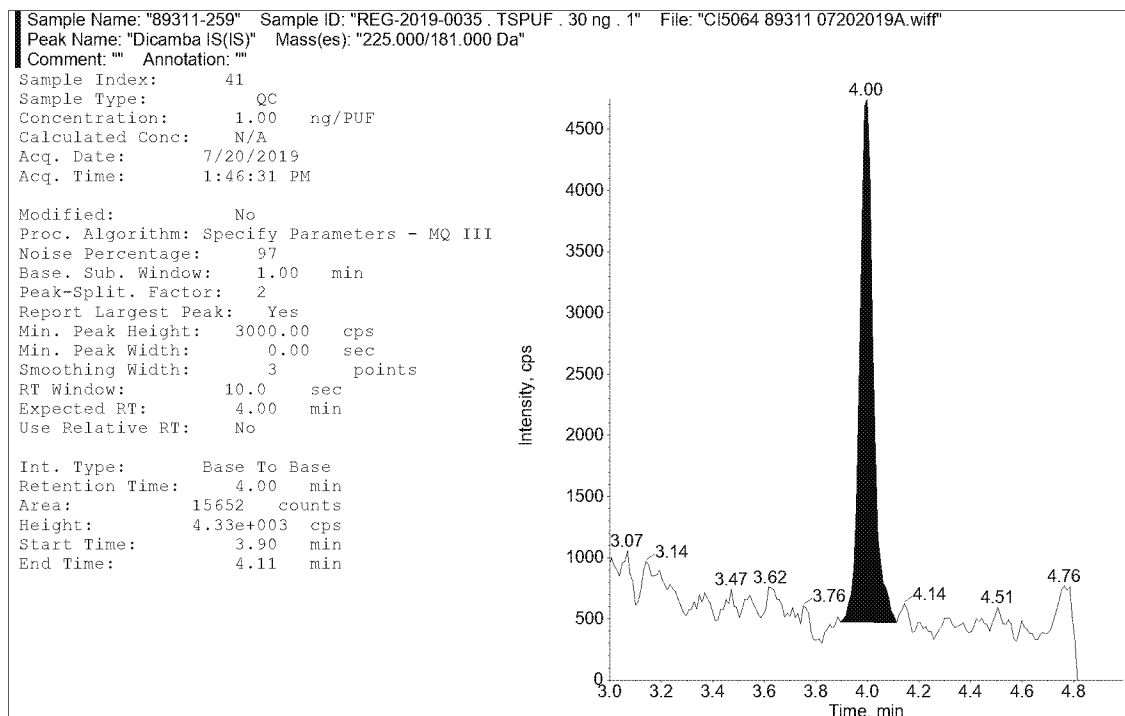
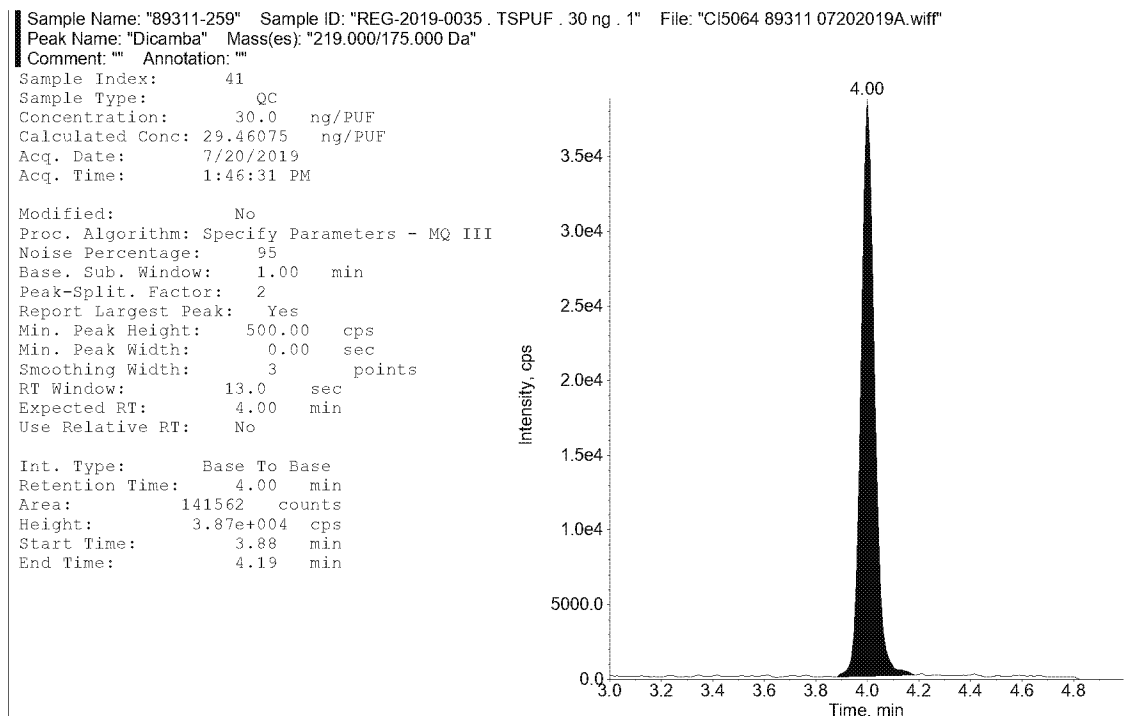
**EEAL ID: 89311-256**      **Sample ID: REG-2019-0035 . TSPUF . 0 ng . 1**  
**Expected Retention Time: 4.00 min.**



Representative Chromatograms from Transit Stability PUF (cont'd)

EEAL ID: 89311-259

Sample ID: REG-2019-0035 . TSPUF . 30 ng . 1



## Appendix M. Analysis Conditions

### Example High Performance Liquid Chromatography Operating Conditions for the Determination of Dicamba on Filter Paper and Tank Mix Samples

Instrumentation: Agilent High Performance Liquid Chromatography (HPLC) system, with Agilent Degasser, Agilent Pump, Agilent Autosampler, Agilent Controller and Column Manager, Agilent Detector, with Empower 3 Software for data collection and system control

Column: Alltima 5 $\mu$ m C18, 250 mm x 4.6 mm

Guard Column: NA

Mobile Phase: Component A: 0.1M Phosphoric Acid (aq)  
Component B: Acetonitrile

Gradient:

<u>Time (min.)</u>	<u>% A</u>	<u>% B</u>
1.00	60.0	40.0
10.00	0.0	100.0
15.00	0.0	100.0
16.00	60.0	40.0
20.00	60.0	40.0

Run Time: 20 minutes

Flow Rate: 1.500 mL/min

Injection Volume: 20  $\mu$ L

Column Temperature: 30  $^{\circ}$ C

Detector Type: UV,  $\lambda$  = 280 nm

**Example Liquid Chromatography Operating Conditions for the Determination of  
Dicamba on PUF Samples**

Instrumentation: Applied Biosystems (AB)/Sciex API 6500+ Q-Trap with SelexION<sup>®</sup>+ Mass Spectrometer LC-MS/MS system with AB/Sciex ExionLC<sup>™</sup> AD System: Solvent Degasser, Pumps, Autosampler, Heated Column Compartment, Solvent Valve, and Controller with AB/MDS Sciex Analyst Software for data collection and system control (version 1.6.3)

Column: Phenomenex Kinetex Biphenyl, 3.0 x 50 mm, 2.6  $\mu$ m

Guard Column: Phenomenex SecurityGuard Biphenyl, 3.0 mm

Mobile Phase: Component A: 0.05% Formic Acid (aq)  
Component B: Methanol

Gradient:

<u>Time (min.)</u>	<u>% A</u>	<u>% B</u>
Initial	80	20
4.00	45	55
4.01	5	95
6.00	5	95
6.01	80	20
7.00	80	20

Run Time: 7 minutes

Flow Rate: 0.500 mL/min

Injection Volume: 10  $\mu$ L (1.0  $\mu$ L for 60.0 ng/PUF fortifications)

Column Temperature: 40  $^{\circ}$ C

### Example Mass Spectrometry Operating Conditions for the Determination of Dicamba on PUF Samples

Acquisition Time:	5 minutes
Polarity:	Negative
Scan Type:	MRM
Resolution Q1:	Unit
Resolution Q3:	Low
Curtain Gas (CUR):	20.00
Collision Gas (CAD):	“Medium”
IonSpray Voltage (IS):	-2500.0
Temperature (TEM):	300 °C
Ion Source Gas 1 (GS1):	70.00
Ion Source Gas 2 (GS2):	60.00
Declustering Potential (DP):	-10.00
Entrance Potential (EP):	-10.00
Collision Energy (CE):	-9.00
Cell Exit Potential (CXP):	-9.00

### Quantitation and Confirmation Transitions Monitored

Analytes:	Precursor Ion Q1	Product Ion Q3	Dwell Time (ms)
Quantitation			
Dicamba	219	175	150
<sup>13</sup> C Dicamba IS	225	181	150
Confirmation			
Dicamba	221	177	150
<sup>13</sup> C Dicamba IS	227	183	150

### SelexION®+ Conditions:

Separation Voltage (SV):	2000.0
Compensation Voltage (COV):	-11.00
DMS Offset (DMO):	50
DMS Temperature (DT):	Low
DMS Resolution Enhancement (DR):	Open
Modifier (MD):	2-Propanol
Modifier Compensation (MDC):	Low
Modifier Density (MDD):	0.79
Modifier MW (MDW):	60.10